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Journal of the Mineralogical Society of America

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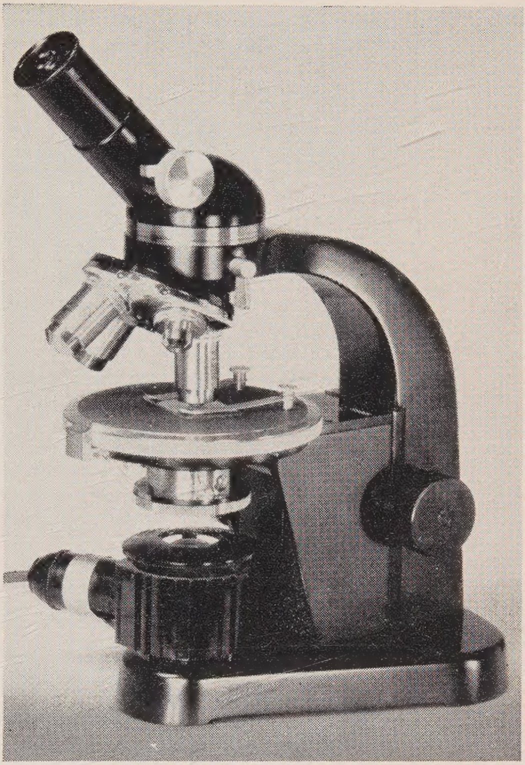

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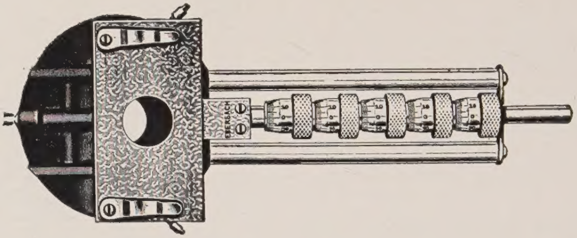

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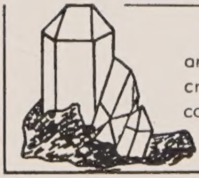

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Vol. 45 MARCH–APRIL, 1960 Nos. 3 and 4

SOME APPLICATIONS OF CLAY MINERALOGY*

RALPH E. GRIM, *Department of Geology, University of Illinois, Urbana, Ullinois.*

INTRODUCTION

Clay materials are the essential components of most soils and therefore, are of great importance in agriculture. The engineer is frequently required to build a structure on, through, or with soil materials. Clays are used as raw materials in many manufactured products, for example, ceramic products, paper, paint, and catalysts. The properties of clay materials are determined largely by their clay mineral composition, or more fundamentally by the structure, composition, and physical attributes of the clay minerals. It follows, therefore, that knowledge of the structure, composition and properties of the clay minerals has great practical value.

This matter of applying knowledge of the clay minerals to practical problems involving the use of clays and soils has fascinated me for a long time. I like to call it "Applied Clay Mineralogy." I wish to present a few examples of the application of clay mineral data mostly from my own experience, but in a few instances from the literature.

There is another reason for selecting this subject for this address. I am convinced that the application of mineralogical knowledge is an excellent way to make known to the general public the importance of our science, and to gain for it, its proper place among the other sciences. It seems to me that too many mineralogists not only have no interest in the application of their findings, but have actually ignored any possible application, to the detriment of the development of mineralogy.

CERAMICS

There are many applications of clay mineralogy to ceramics but one example will suffice. In the manufacture of many ceramic products, clays are heated to elevated temperatures to develop strength, permanence, desired color, etc. The ceramist wishes to fire his product with the least possible cost which involves the use of the least fuel and in the shortest possible time.

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* Address of the retiring President of the Mineralogical Society of America at the 40th meeting of the Society at Pittsburgh, Pennsylvania, November 3, 1959.

Equilibrium phase diagrams have been of great value to the ceramist. However, they do not always provide the data he needs. Thus the ceramist is interested in the events that take place in the clay at low temperatures long before equilibrium is reached and in the rates at which the various changes take place on heating. He is interested in the effect of traces of elements on high temperature reactions. Studies of the changes taking place when the clay minerals are heated from ordinary temperatures up to their fusion point by differential thermal analyses and by continuous *x*-ray diffraction procedures have recently provided data of tremendous importance to the ceramist.

When the clay minerals are heated to temperatures of the order of 400° C. to 600° C. they lose their hydroxyl water. It has long been thought necessary to fire clays through this temperature interval very slowly. Studies of the dehydration characteristics of the clay minerals have shown that for some clay minerals the dehydration reaction is abrupt and that it cannot be slowed down. Further, in many instances the dehydration is accompanied by no complete breakdown of atomic structure or shrinkage. This is not true for all clay minerals, but for many of them there is no reason to go through this temperature interval slowly when clay products are fired. Slow heating in the temperature interval is often a waste of time and fuel (Grim and Johns, 1951).

In the manufacture of many refractory bricks it is desired to burn so as to produce the maximum amount of mullite. It has been shown (Grim and Kulbicki, 1957), that when kaolinite, which is the clay mineral component of clays used in the manufacture of such brick, is heated to about 950° C.–1000° C. some mullite is formed, but it is not until about 1250° C. that the mineral develops rapidly. Heating in the interval 950° C–1250° C. will not develop much mullite when kaolinite is fired, even if the temperature is maintained for very long periods of time. Therefore, slow heating in this interval is useless. It has been shown further by Wahl (1958) that the addition of traces of various elements can enhance or retard the development of mullite at the higher temperature but that such additives have very little if any effect on the formation of mullite at the lower temperature.

In summary then, fundamental studies of the changes taking place when the clay minerals are heated have been of great practical value by showing the ceramist how to arrange his firing schedule for the most economical burning of his products. Indeed, the matter has been carried further in the development of many refractories which are not fired until they are placed in service. Unfired brick or plastic material used to line furnaces or ladles is in effect burned extremely rapidly when first used.

ENGINEERING

The usual procedure in engineering practice when soil materials are involved in construction is to obtain empirical test data on such properties as compaction under load, plasticity, etc., of the soils involved. The design of the structure is based on such test data, evaluated somewhat on the basis of past experience. In the actual construction, the environmental setting of the soil may be changed and with it the properties of the soil so that the test data may need re-evaluation. In essence the engineer frequently must predict the changes in soil properties when he changes the environment. Engineers are coming more and more to appreciate this situation and are becoming interested in the fundamental factors determining the properties of soils realizing that this is important if they are safely to predict the changes that may take place in the soil when they place a load on it, change the water table, place in it a mass of concrete providing a flood of calcium ions for cation exchange reaction, etc. This interest is attested by the symposium of the American Society of Civil Engineers held last year on clay mineralogy in relation to soil mechanics and by the symposium of the Highway Research Board of the National Research Council this year on the properties of soil-water systems.

I would like to present several examples of the relation of clay mineralogy to engineering problems.

The clay mineral halloysite occurs as $2H_2O$ and $4H_2O$ hydrates. The $4H_2O$ form dehydrates to the lower hydration form at ordinary temperatures slowly and irreversibly passing through an intermediate state of hydration. Neither the $2H_2O$ or $4H_2O$ form develops any substantial plasticity when mixed with water as most other clay minerals do. However, the intermediate state is likely to be very plastic and has physical properties quite unlike either the $2H_2O$ or $4H_2O$ forms. A test of properties, therefore, of a soil containing the $4H_2O$ form under conditions such as the engineer generally uses in obtaining his test data which prevent the loss of any water, probably will not give data showing the properties of the soil under conditions of actual use when some moisture may be lost. Failure to appreciate this characteristic of halloysites can lead to serious difficulties for the engineer. Fortunately halloysite is not a very common constituent of soils, but it is very desirable to detect the presence of halloysite in any soil that may be part of a construction program.

There are in some parts of the world soils which have considerable bearing strength in their natural undisturbed state, but very little or no strength in the remolded state, i.e. after their original texture has been destroyed. This is the property called sensitivity by the engineer. Extreme examples are the so-called "Quick" clays well known in many countries but especially in Norway where they have been intensively studied by Rosenquist (1953) and Bjerrum and Rosenquist (1956). These investigators have been able to produce "Quick" clays in the laboratory and have demonstrated the reason for their unusual properties.

It must be emphasized that the explanation offered for these "Quick" clays does not account necessarily for the high sensitivity of other soils. There are other explanations for some other examples.

The clay mineral component in the Norwegian "Quick" clays is largely illite. Some quartz is present also, and much of the material in the crude clay is in a coarse clay or fine silt particle-size range. The material was deposited under marine conditions in a flocculated condition with a loose open texture and with sodium as the dominant adsorbed and exchangeable cation. Later, under present conditions the sodium has been leached being replaced by other cations such as hydrogen, without any change in the natural moisture content of the soils. The water adsorbed directly on the surfaces of the clay minerals is not liquid water but is composed of oriented water molecules and it therefore, serves as a bond, helping to hold the clay mineral particles together and to give strength to the soil. When sodium is present, adsorbed on the surface of the clay mineral particles, the oriented water develops to a very considerable thickness, that is, many molecular layers in thickness. With other adsorbed cation the oriented water does not develop to thicknesses as great as when sodium is present. In these clays the adsorbed water developed initially in equilibrium with sodium. In a sense the sodium ion acted as a kind of prop to maintain the rigid water and to give strength to the soil. The leaching of the sodium ion removed the prop and did not at the same time change the amount of water. There was, therefore, a loss of equilibrium and a condition of great instability. Any slight movement or jarring of such a soil is likely to change the rigid water to fluid water with a complete loss of strength. This is what happens in the sudden development of landslides in "Quick" clay.

An understanding of "Quick" clays, therefore, involves an understanding of the nature of the water adsorbed by the clay minerals and the way adsorbed ions affect this water. This is a matter of very great interest at the moment and much attention is being given to investigations of the nature of the water in clay-water systems. The approach of colloid chemistry to investigations of clay-water systems has paid particular attention to the forces of attraction and repulsion between the particles. The water was thought to have a passive role and to do very little more than provide a medium to separate the particles and to provide for their hydration. Structural attributes have now entered the picture—the structure of the water in its relation to the structure of the clay minerals—particularly in systems where the relative amount of water is not large as is the case in the plastic state.

To pass on to another example in the field of engineering. Some years years ago it was desired to construct a fresh water lake on Treasure Island in the San Francisco Bay area in the preparation for a World's Fair, (Lee, 1940). A depression was made and lined with a plastic clay and fresh water was pumped into it. The fresh water seeped away rapidly. The leakage of the depression could have, of course, been stopped by the placement of an impervious seal such as concrete or asphalt, but a less expensive solution was much desired. An astute engineer knowing something of the composition of the soil and the properties of clay minerals in general suggested that sea water be pumped into the depression and after standing for some time it be removed and replaced with fresh water. This was done and the leakage was stopped. The explanation is that the soil contained some montmorillonite which is a very dispersible clay mineral. The presence of the sea water led to an exhange reaction whereby sodium became the adsorbed cation. The sodium ion caused enhanced dispersion of the clay mineral, that is, it caused it to break down into more and smaller particles. Further the sodium ion led to the development of thick adsorbed layers of oriented water. For both of these reason the sodium clay acted as a seal for the water whereas the clay mineral with another cation did not act as such a seal.

PETROLEUM INDUSTRY

Clay mineral studies are of wide interest to the petroleum industry; in correlation problems, in propsecting, in drilling muds, in secondary recovery operation, in refining processes, etc. I wish to consider here just one of these applications and that in the less generally known field of catalysis in the refining operation.

It has been known for a long time that some of the clay minerals have catalytic properties towards many organic compounds. This property may be desirable or detrimental, in the use of clays in the petroleum industry. It is desirable in the use of clays as catalysts in the manufacture of gasoline from crude oil. It is undesirable when the clay is used to decolorize or purify a product, and it is desired that the product shall not be changed in any way. It is very undesirable when clays are used as carriers for insecticides made from hydrocarbons where the catalytic action may rapidly cause a loss of toxicity of the insecticide.

In recent years there has been much research in many laboratories directed towards finding ways to modify or control the catalytic properties of the clay minerals and this has led to studies of the fundamental factors which give the clay minerals their catalytic properties. Although much remains to be learned on this matter, some generalities appear to be established (for an introduction to this subject, (see Mathieu, 1945; and Milliken, Oblad, and Mills, 1955). Thus, the structure of the clay minerals is important, particularly the structure of the surface of the tetrahedral silica sheet part of the structure. When the silica tetrahedral sheet structure is lost the catalytic activity is lost. The surface of the silica tetrahedral layers must have the property of donating protons and it must be substantially clean of adsorbed alkali earth cations and probably other ions. Further, some aluminum is essential, probably in some particular pattern behind the silica surface.

The problem in preparing a catalyst is to make one that produces the proper products, for example, a large percentage of high octane gasoline from a given feed stock. The catalyst can be too active in that it will break down the hydrocarbon completely to a gas and carbon. Also the catalyst must be hard and resistant to abrasion. It must be durable so that it will withstand regeneration through many cycles of use. These conditions have been met, and now catalysts are produced from clays composed of montmorillonite, halloysite, and kaolinite. Essentially the conditions have been met by processes which modify the structures of the clay minerals but do not destroy them. The character of the catalyst is controlled by controlling the modification of the structure. I would emphasize that success has been obtained by looking at the problem through "structural glasses" rather than simply on the basis of the amount of the elements present.

Bonding Clays

In the casting of metals in foundries, synthetic mixtures of sand and clay are used as a medium for the preparation of the molds into which the molten metal is poured. Various types of clays are used each giving fairly distinctive properties, depending on the clay mineral composition and the exchangeable cations that are present (Grim and Cuthbert, 1945, 1946). Thus, bentonite from Wyoming, which is composed of montmorillonite carrying sodium as the exchangeable cation provides very high dry strength and moderately high green strength, i.e., strength before drying to remove the tempering water. Bentonite from Mississippi, composed of montmorillonite, which carries calcium and hydrogen as exchangeable cations provides very high green strength and moderately low dry strength. So-called fire-clays which are underclays of Carboniferous age produced extensively in Middle Western states and composed of poorly ordered kaolinite and illite provide good flowability, durability, and good metal surface characteristics. The fireclays have lower green strength than the bentonites, but their geographic location close to foundries makes it economic to use a larger proportion of them in the sand which in turn enhances their desirable properties.

A third type of clay used in foundries is an illite which is very poorly ordered and has a small amount of mixed-layer montmorillonite (Grim and Bradley, 1939). This interesting clay was discarded as worthless overburden by a refractory manufacturer until a clay mineral analysis suggested its potential use as a bonding clay. It has strength characteristics intermediate between those of the sodium and calcium montmorillonites and the flowability and durability of the kaolinite clays. One of the problems sometimes encountered by foundries is the development of many very small pits or depressions in the surface of the cast metal which are known as pin holes. Recently Jones and Grim (1959) found that when illite clay is used such pin holes do not develop. The explanation seems to be that the tempering water and hydroxyl water of this type of clay is gradually driven off when metal is poured into the mold, rather than abruptly as is the case for some other clays. Also the clay is very thoroughly dispersible throughout the sand so that there are no concentrated masses of clay with adsorbed water to suddenly release relatively large amounts of water vapor at given spots to produce the pin holes.

Sometimes it is not possible to translate a laboratory finding that appears to have great practical value into economic usage. For example, some years ago a study of the bonding properties of various types of clay minerals showed that a sample of halloysite had excellent strength characteristics and provided castings with an excellent smooth surface. Halloysite clay had never been used in foundries before, and it appeared to be a discovery of considerable importance. The sample tested had been in the laboratory for some time and was in an intermediate state of hydration between the $2H_2O$ and the $4H_2O$ form. Studies of potential sources of halloysite showed that the material back from exposures was the $4H_2O$ form and that any tonnage production would have to be the $4H_2O$ form which unfortunately has substantially no bonding strength. So far it has been impossible to slightly dehydrate the $4H_2O$ form in the laboratory in any way that could be used commercially to produce the high strength material. Apparently only slow loss of water from the $4H_2O$ form produces the high strength material.

PAPER

The manufacture of paper requires huge tonnages of clay as filling and coating agents to make the paper opaque, white, and printable. For these purposes large tonnages of substantially pure kaolinite clays are used. Actually about two-thirds of all of the kaolin produced goes into the paper industry. Such kaolins are produced extensively from sedimentary beds of Upper Cretaceous age in the southeastern part of the United States and from clays of hydrothermal origin in the Cornwall district of Great Britain.

To obtain the necessary purity and whiteness, the clays are treated chemically to remove various iron compounds. The clays are then washed with wet sedimentation procedures to remove quartz and other non-kaolinite particles. It is well known that the size of the kaolinite particles is important in determining the properties of the coated paper and fractionation is now practiced using centrifuges to produce kaolins of closely controlled particle size in the micron size range. Murray and Lyons (1956) have shown recently that the shape and crystal perfection of the kaolinite particles is important as well as their size in determining coating characteristics. Well crystallized kaolinite particles with distinct hexagonal outlines in book-like units which are not too thin are required.

In the paper coating operation the clay is made into a slurry which is flowed on to the paper stock as it moves through the paper making machine. The paper stock moves at a very rapid speed and consequently clay slurries of the highest possible concentration at the lowest viscosity without any dilatency are required. These specifications pose problems. There are kaolins which give slurries of low viscosity, and other kaolins which produce viscosities several times as great with the same clay concentration, which do not seem in any way to differ from the low viscosity kaolins. *X*-ray diffraction and electron micrographic analyses reveal no differences in the composition of the clay or in the size and shape of the kaolinite particles. The cause of the differences is unknown, but it must reside in some factor of the surface characteristics of the kaolinite particles or in their shape which is not revealed by the electron micrographs. In some cases viscosity differences can be explained by the presence of small amounts of montmorillonite, but in other cases the differences are shown by pure kaolinite kaolins.

ORGANIC-CLAD CLAY MINERALS

One of the areas of most intensive research in clay mineralogy in recent years has been the study of clay mineral-organic reactions (for an introduction to this subject see Grim, 1953). It has been shown that the clay minerals can enter into cation exchange reactions with organic cations in which case the organic molecule is held on the clay mineral surface by a coulombic bond. The clay minerals can also adsorb highly polar organic molecules which are held mainly by hydrogen bonds. VanderWaal's forces also play a role in attaching the organic molecule to the clay mineral surface.

These reactions permit the coating of the clay mineral surfaces with organic molecules producing so-called organic-clad clay mineral particles which in general have been changed from hydrophilic to hydrophobic. Such organic-clad clay minerals become oleophilic in that they can be dispersed in organic liquids and some organic molecules can be polymerized to the organic molecules coating the clay mineral surface. In the case of the exchange reactions, by knowing the surface area per exchange position one can select an organic molecule that completely coats the surface or one that only partially covers the surface. Thus, the clay mineral can be made partially hydrophobic by selecting an organic molecule of the proper size and shape. This is assuming, of course, that one knows the orientation of the organic molecule on the clay mineral surface. Also by selecting the proper coating molecule the clay mineral particles can be made particularly reactable with some types of organic vehicles.

It it interesting that researches in this field started out as a purely fundamental study of clay mineral-organic reactions, and that they have led to completely new clay products which have opened new uses for clays. Organic-clad montmorillonites and kaolinites are now produced commercially. New uses of these products are in the field of gelling agents in the manufacture of lubricants, as fillers and strengthening agents in plastics, as components of printing ink, as flattening agents in paints and many others. In some cases the organic-clad clay mineral is "tailor made" to fit a particular organic material with which it is to be used.

Another area of the application of clay mineral organic reactions is worth notice. That is, in conditioning soils for agricultural use and in stabilizing them for the construction of roads and airport runways (Winterkorn, 1946). In such cases an organic agent is added to the soil and the hoped for effect of the additive must be obtained through a reaction with the clay minerals in the soil. The hoped for results have not always been attained, frequently because the investigators forgot or did not know that various soils contain different clay minerals and that the reaction of organic molecules was not the same for all types of clay minerals.

DISPOSAL OF ATOMIC WASTE

An area of clay mineral research of extreme importance at the moment is the possible use of clays in the disposal of atomic wastes from nuclear reactors. Some procedure for the disposal of hot isotopes with a long half life must be discovered that is safe and economical before there can be widespread development of power from such reactions. One method that seems to have much promise is to adsorb the isotope by clays and then fire the clay to lock up the hot element in an insoluble compound so that the fired material can be buried safely without the risk of the isotope going into solution and contaminating the ground water.

Clay minerals with high cation exchange capacity and low fusion temperatures seem to be desirable and bentonites carrying montmorillonite with cation exchange capacities of about 130 milliequivalents per 100 grams have been suggested (Kerr, 1959). It appears, however, that cation exchange capacity is not the sole determining factor. For example, some of the waste liquors are highly acid and have a high electrolyte content. The property of attapulgite-water systems of being relatively unaffected by large variations in the electrolyte content has indicated that this type of clay may be desirable in some instances.

Before final disposal it may be desirable to separate and isolate some of the isotopes for which there is a possible use. This has led to the investigation of the selective adsorptive properties of various clay minerals with the hope that they could be used as atomic sieves. Barrer and his colleagues in Great Britain (1954) have done much work in this field. It is too early to tell if clay minerals can be used as such atomic sieves. Possibly the clay mineral structures can be altered as in the case of the manufacture of clay catalysts to develop specific adsorptive properties. At the present time the atomic sieves in use are synthetic zeolites. It is worth pointing out that the research that led to the development of such atomic sieves is essentially mineralogic.

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LOUGHLINITE, A NEW HYDROUS SODIUM MAGNESIUM SILICATE[1]

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ABSTRACT

A new hydrous sodium magnesium silicate from the Green River formation in Sweetwater County, Wyo., having the formula $Na_2O \cdot 3MgO \cdot 6SiO_2 \cdot 8H_2O$, is named after Dr. Gerald F. Loughlin, former Chief Geologist of the U. S. Geological Survey.

The mineral is found in dolomitic oil shale and in thin section has been seen replacing shortite, northupite, and searlesite.

Loughlinite is a fibrous, pearly-white mineral with a silky luster and is virtually identical to sepiolite ($4MgO \cdot 6SiO_2 \cdot 8H_2O$), both in hand specimen and microscopically. Loughlinite may be distinguished from sepiolite by the 8 per cent Na_2O in the structure and by comparison of the x-ray fiber diagrams. The indices of refraction of loughlinite are $\alpha = 1.500$, $\beta = 1.505$, and $\gamma = 1.525$.

Prolonged leaching of loughlinite with water or immersion of loughlinite in magnesium salt solutions converts the mineral into a material that is apparently identical to sepiolite.

INTRODUCTION

During the logging of the drill core of the John Hay, Jr., Well No. 1, Sweetwater County, Wyoming, in 1940, in which the new minerals shortite (Fahey, 1939) and bradleyite (Fahey and Tunell, 1941) were found, a new asbestos-like mineral, a hydrous sodium magnesium silicate, was found in quantities too small for a thorough study. The same mineral was observed in 1946 in the drill core of the nearby Union Pacific Well No. 4, but here too in sparse amounts. However, in the summer of 1947, many specimens of dolomitic shale containing this fibrous mineral were obtained from material excavated during the sinking of the shaft of the Westvaco Chlorine Products Corporation that was put down for the purpose of mining trona. These three locations are between 18 and 20 miles west of the town of Green River, Sweetwater County, Wyoming. It was not until recently that sufficient material was purified for chemical analysis and related studies.

Loughlinite (Fahey and Axelrod, 1948) was first announced in a paper given at the 28th annual meeting of the Mineralogical Society of America that was held in Ottawa, Canada, in December 1947. The name loughlinite has been given to the new mineral in honor of the late Dr. Gerald F. Loughlin, who, during his many years as Chief Geologist of the U. S. Geological Survey, did much to encourage research in mineralogy.

PHYSICAL PROPERTIES

Loughlinite is a fibrous, pearly-white mineral with a silky luster, and is

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[1] Publication authorized by the Director, U. S. Geological Survey.

TABLE 1. CHEMICAL ANALYSES

	Loughlinite (leached 6 hrs.)			Loughlinite (leached 58 days)		Sepiolite	
	A	A′	Molecular ratios	B	Molecular ratios	C	Molecular ratios
SiO_2	50.80	50.80	6.00	51.52	4.97	52.50	6.00
Al_2O_3	0.66	0.66	0.05	0.75	0.04	0.60	0.04
Fe_2O_3	1.85	1.85	0.08	2.64	0.10	2.99	0.13
FeO	1.51	1.51	0.15	1.35	0.11	0.70	0.07
TiO_2	0.02	0.02	0.00	0.02	0.00	—	—
CaO	0.12*−0.12=	0.00	0.00	0.02	0.00	0.47	0.06
MgO	16.18 −0.17=	16.01	2.82	19.60	2.82**	21.31	3.63
Na_2O	8.16	8.16	0.93	0.06	0.01	—	—
K_2O	0.00	0.00	0.00	0.00	0.00	—	—
$H_2O-110°$ C.	13.68	13.68	8.08	13.38	7.66		
$H_2O+110°$ C.	6.82	6.82		10.42		21.27	8.10
CO_2	0.30*−0.30=	0.00	0.00	0.00	0.00	—	—
	100.10 dolomite	0.38		99.76		99.84	
	magnesite	0.21					
		100.10					

A Loughlinite (leached 6 hrs.), J. J. Fahey, analyst.
A′ Loughlinite (leached 6 hrs.) after deduction of dolomite and magnesite.
B Loughlinite (leached 58 days), J. J. Fahey, analyst.
C Sepiolite from Ampandrandava, Madagascar (Caillère, 1951, p. 230).
* Calculated as dolomite and magnesite.
** The molecular ratio of MgO is arbitrarily given the value 2.82.

virtually identical in appearance to sepiolite, $4MgO \cdot 6SiO_2 \cdot 8H_2O$, both in a hand specimen and microscopically. The physical and optical constants were determined, and the chemical analysis was made (Table 1, Sample A) on material that had been leached for 6 hours to remove contaminating sodium carbonate, and air dried at room temperature for 10 days.

The specific gravity of loughlinite is 2.165. This was measured by means of a fused silica Adams-Johnston pycnometer, using distilled water. Owing to the extreme fineness of the fibers no effort was made to determine hardness. The indices of refraction of loughlinite are:

$$\alpha = 1.500$$

$$\beta = 1.505$$

$$\gamma = 1.525$$
$$2V = 60° \text{ positive (calculated)}$$

The indices were measured on randomly oriented bundles of tiny fibers. By making very many measurements a close approximation of the true value of α, β, and γ is believed to have been made. Loughlinite has parallel extinction and positive elongation. A thin section cut normal to the long axis of a bundle of the fibers gave a positive acute interference figure. No twinning was observed.

Loughlinite which had been leached for 58 days (Table 1, analysis B) was so matted and finely divided that only $\alpha = 1.520$ and $\gamma = 1.532$ could be measured.

Differential thermal analyses of the natural and the treated loughlinites have been made by our colleague George T. Faust who will report elsewhere on his studies. His data show that loughlinite gives a DTA curve similar to that of sepiolite. The studies of Caillère and Henin (1947) showed that the DTA curves of sepiolite and palygorskite could be altered by treatment with various salt solutions previous to thermal analysis. The leaching experiments performed in the study in purifying the mineral are equivalent to treatments in salt solution.

PARAGENESIS

Loughlinite is found in medium- to low-grade oil shale of the Wilkins Peak member of the Green River formation. It was formed after compaction of the sediments had taken place, as is indicated by the absence of flow lines in the shale. Loughlinite is found in veins, usually less than 3 cm long, that vary in thickness up to 1 cm, which is the maximum length of the fibers.

In thin section, it is readily seen that loughlinite replaces the dolomitic shale, isolated small islands of the shale being frequently observed within the area occupied by loughlinite. Shortite ($Na_2CO_3 \cdot 2CaCO_3$), northupite ($Na_2CO_3 \cdot MgCO_3 \cdot NaCl$), and searlesite [$NaB(SiO_3)_2 \cdot H_2O$] are likewise seen being replaced by loughlinite. In no instance has loughlinite been observed in the process of replacement by another mineral.

CHEMICAL PROPERTIES

The sample was prepared by carefully handpicking the fibrous mineral from vugs opened up on freshly broken surfaces of the dolomitic shale. It was not necessary to use a binocular microscope in this operation, except in the final examination of the picked material. Care was taken to free the ends of the fibers from small particles of adhering shale. All shale except a quantity estimated at less than one per cent was finally removed by agitating, with a glass stirring rod, the fluffy fibrous material in a large beaker. After about 20 minutes of this treatment the loughlinite was removed, leaving fine particles of shale in the bottom of the beaker. This operation was repeated several times until no residual shale remained in the beaker.

Wilkins Peak member of the Green River formation, wherein loughlinite is found, contains throughout its vertical column, varying small quantities of sodium carbonate, so finely disseminated as to be undetectable by means of the petrographic microscope, even when using an oil-immersion objective. Addition of a drop of acid confirmed the presence of carbonate in the prepared sample of loughlinite, and a quantitative determination showed 1.72 per cent CO_2 to be present. Electron diffraction powder patterns indicated that a small amount of extraneous material was present in the prepared loughlinite sample. This extraneous material could not be positively identified.

Most of the carbonate was removed by placing the fluffy asbestos-like material (approximately 17 grams) in a one-liter Erlenmeyer flask, adding about 700 ml. of distilled water at room temperature, shaking well and allowing to remain for 6 hours. After filtering with suction on a Buchner funnel the sample was air-dried for about 10 days. The mat that had formed on the funnel was then shredded to its former fluffy condition, allowed to air-dry for another week, and was then ready for analysis (Table 1, analysis A).

Analysis A shows that some carbonate is still present after 6 hours of leaching. It is known that a small amount of shale containing dolomite and magnesite is present in the prepared sample. The residual 0.30 per cent CO_2 was then assumed to be from these contaminants.

The x-ray fiber diagrams of loughlinite taken before and after the 6-hour leaching process are identical.

It was found that prolonged leaching at room temperature gradually removed sodium and probably silica from loughlinite. A sample of approximately 12 grams of the analyzed sample of loughlinite (Table 1, analysis A) was placed in a 1 liter Erlenmeyer flask, 200 ml. of distilled water was added, and after shaking well, it was allowed to remain (stoppered) for 5 days at room temperature. The water was then filtered off, and the sample air-dried. Upon analysis the per cent Na_2O was found to have dropped from 8.16 to 6.96. The sample was then returned to the flask and 200 ml. of water added. The flask was stoppered, well shaken, and allowed to remain for an additional 8 days, or a total of 13 days of leaching. The analysis of the sample after filtering and air-drying showed that the Na_2O content had been reduced to 5.60 per cent. This leaching process was repeated for an additional 45 days, making a total leaching time of 58 days. The leached and dried sample then contained only 0.06 per cent Na_2O (Table 1, analysis B), and gave an *x*-ray fiber diagram identical to that of sepiolite.

A comparison of analysis A′ with analysis B shows that the Mg/Si ratio increased upon leaching for 58 days. It is apparent then that either Si was removed or that Mg was added to the loughlinite structure during this leaching process. It is improbable that magnesium was added to the structure since only distilled water (approximately 2000 ml.) was used in the leaching process. It was also noted that colloidal silica was present in some of the wash water after leaching.

A sample of loughlinite was treated for 7 days in a small amount of 1 per cent $MgCl_2$ solution and was then washed with a small amount of distilled water. The resulting material gave an *x*-ray fiber diagram identical to that of sepiolite. This experiment, later repeated with 200 ml. of 2 per cent $Mg(NO_3)_2$ solution, gives the same results.

Samples of loughlinite which had been leached for 58 days or which had been treated with a 1 per cent $MgCl_2$ solution for 7 days, were treated with cold 30 per cent NaCl solution for 11 weeks. Similar samples were treated for 9 days with a cold saturated solution of Na_2CO_3. Neither treatment resulted in a reversion to the original loughlinite as shown by the *x*-ray fiber diagrams.

The 13.68 per cent of water lost from loughlinite at 110° C. (Table 1, analysis A) was completely regained after 8 hours at room temperature and humidity.

Under similar conditions sepiolite regains about 80 per cent of the water lost at 110° C.

Loughlinite is differentially soluble in hydrochloric and sulfuric acids, leaving a siliceous skeleton that retains the shape of the fibers. When heated to 900° C. incipient fusion takes place.

X-ray Studies

Three types of *x*-ray patterns were made in this study. These are:

1. *X*-ray fiber diagrams taken in a rotation camera by rotating a small bundle of fibers about the fiber axis (*c*). These patterns show the hk0, hk1, hk2, hk$\bar{1}$ and hk$\bar{2}$ nets of loughlinite and sepiolite.

2. *X*-ray zero-level fiber diagrams taken in a powder camera with a radius of 57.3 mm. These patterns show only the hk0 net of loughlinite and sepiolite.

3. *X*-ray powder patterns. In an attempt to minimize the preferred orientation the *x*-ray powder spindle was made from fibers that had been rolled into a ball mount (Hildebrand, 1953).

X-ray fiber diagrams taken in a rotation camera show the following:

1. Untreated loughlinite is identical to loughlinite (leached 6 hours).
2. Loughlinite (leached 6 hours) definitely has a different unit-cell than that of sepiolite from Ampandrandava, Madagascar.
3. Loughlinite (leached 58 days) is identical to sepiolite.
4. Loughlinite (leached 6 hours then treated 7 days in $MgCl_2$ solution) is identical to sepiolite.

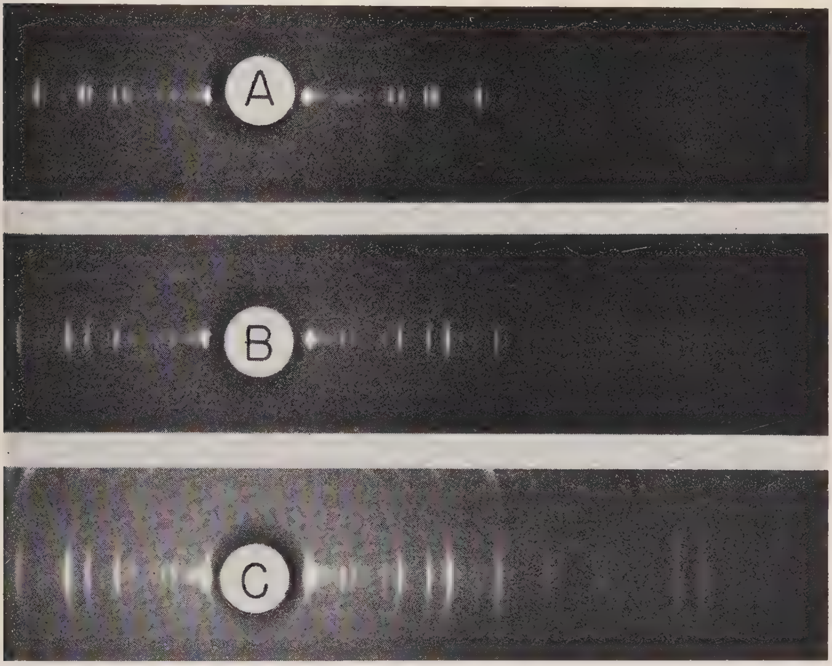

FIG. 1. Zero-level fiber diagrams of
A) loughlinite, Sweetwater Co., Wyo. (leached 6 hrs.), Table 1, analysis A.
B) sepiolite, Ampandrandava, Madagascar, Table 1, analysis C.
C) loughlinite, Sweetwater Co., Wyo. (leached 58 days), Table 1, analysis B.

5. The dimension along the fiber direction (*c*) is 5.3 ± 0.1 Å for both loughlinite and sepiolite.

To further illustrate the similarities and differences of loughlinite (leached 6 hours), loughlinite (leached 58 days), and sepiolite, and also to obtained more accurate values for the spacing of the hk0 reflections, zero-level fiber diagrams were made in an *x*-ray powder camera using copper radiation. The patterns of these materials can be seen in Fig. 1. The spacings of the hk0 reflections measured from these three patterns are tabulated in Table 2. These measurements definitely show that

TABLE 2. *X*-RAY DATA MEASURED FROM THE ZERO-LEVEL (l=0)
Fiber diagrams† of loughlinite and sepiolite
CuKα (Ni filter), $\lambda = 1.5418$ Å, cut off at $d_{\text{obs.}} = 13$ Å.

Loughlinite Sweetwater County, Wyo. (Leached 6 hours) (Table 1, analysis A)		Sepiolite Ampandrandava, Madagascar (Table 1, analysis C)		Loughlinite Sweetwater County, Wyo. (Leached 58 days) (Table 1, analysis B)	
d(Å)	I*	*d*(Å)	I*	*d*(Å)	I*
12.8	≫100	12.0	≫100	12.0	≫100
10.2	4	9.6	9	9.6	9
7.6	35	7.5	50	7.5	50
6.4	13	6.7	13	6.7	25
—	—	—	—	6.0	2
5.01	18	5.02	25	5.02	25
4.80	50	—	—	—	—
4.45	100	4.52	100	4.52	100
4.28	6	4.44	4	4.40	6
3.79	100	3.75	70	3.75	70
3.65	100	3.46	6	3.45	6
3.59	35	3.37	70	3.34	100
3.08	13	—	—	—	—
2.90	70	2.94	4	—	—
2.82	6	—	—	—	—
2.77	4	2.69	35	2.68	35
2.56	13	2.59	2	2.57	5
2.44	2	2.42	3	2.40	5
—	—	2.25	13	2.24	9
2.13	4	2.14	6	2.14	6
2.08	1	—	—	2.06	2
1.90	13	1.92	4	1.91	6
—	—	1.88	3	1.87	6
—	—	1.82	3	1.81	6
—	—	1.73	2	—	—
1.668	9	1.690	2	1.694	2
1.644	2	1.642	2	1.641	2
1.623	4	—	—	—	—
—	—	1.594	2	—	—
—	—	1.581	18	1.583	25
1.559	18	—	—	—	—
—	—	1.503	13	1.506	18
1.484	6	—	—	—	—
1.463	4	—	—	—	—
1.414	3	—	—	1.415	2
1.383	3	—	—	—	—
		1.372	3	1.370	6
		1.352	4	1.344	6

* Intensities were estimated visually with a calibrated intensity strip.

† Fiber diagrams were taken in an *x*-ray powder camera, radius=57.3 mm.

loughlinite (leached 6 hours) has a different unit-cell than that of sepiolite whereas loughlinite (leached 58 days) has a unit-cell identical to that of sepiolite.

A zero-level fiber diagram was also made of loughlinite (leached 6 hours) which had been immersed in 200 ml. of 2 per cent $Mg(NO_3)_2$ solution for 10 days. This pattern is identical to the zero-level fiber diagram of sepiolite shown in Fig. 1B.

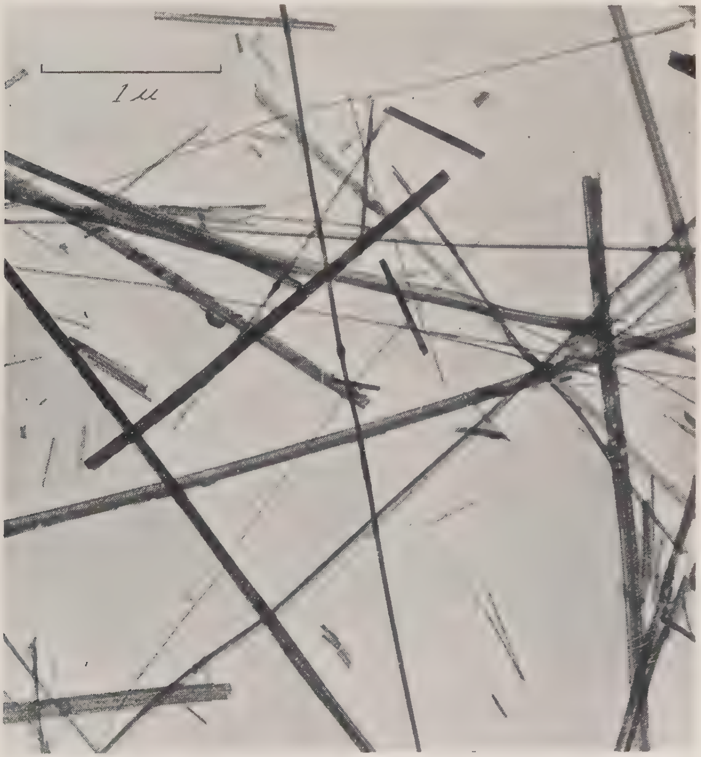

Fig. 2. Electron micrograph of loughlinite (leached 6 hrs.).

The *x*-ray powder data of loughlinite (leached 6 hours), loughlinite (leached 58 days), and sepiolite are compared in Table 3. These powder patterns are somewhat similar in appearance and are not as diagnostic of phase differences as are the fiber diagrams. The differences in the powder patterns of loughlinite (leached 6 hours) and for the sepiolite from Ampandrandava indicate that they are not the same mineral. The powder pattern of loughlinite (leached 58 days) and that of sepiolite from Ampandrandava show no greater differences than are found in

TABLE 3. MEASURED *X*-RAY POWDER DATA FOR LOUGHLINITE AND SEPIOL
CuKα (Ni filter, $\lambda = 1.5418$ Å; cut off at $d_{\text{obs.}} = 13$ Å

Loughlinite Sweetwater County, Wyo. (Leached 6 hrs.) (Table 1, analysis A)		Sepiolite Ampandrandava, Madagascar (Table 1, analysis C)		Loughlinite Sweetwater County, Wyo. (Leached 58 days) (Table 1, analysis B)	
d (Å)	I*	*d* (Å)	I*	*d* (Å)	I*
12.9	100	12.3	100	12.3	100
7.63	5	7.56	8	7.56	3
6.46	1*b*	6.73	4	6.73	1.5*b*
—	—	—	—	6.11	1
5.04	1.5	5.05	6	5.02	3*b*
4.81	5	4.81	<1	4.78	1.5
4.51	7*b*	4.54	14	4.53	6
4.34	18*n*	4.32	13	4.32	18*n*
4.15	1.5	—	—	4.17	2
4.00	1	—	—	3.99	1.5
3.83	6*b*	3.77	14	3.77	9
3.63	7*b*	3.55	3	3.55	3
3.31	5	3.36	15	3.36	6
3.21	1	3.20	8	3.20	5*b*
3.09	2	3.06	3	3.05	1.5*b*
2.92	5	2.84	2*b*	2.84	2*n*
—	—	2.70	6*b*	2.69	2*b*
2.64	6	2.63	6	2.63	3
2.60	5	—	—	2.59	6*n*
2.55	7*n*	2.57	18	2.56	9*n*
2.48⎫	5⎫	2.45	6*b*	2.45	6
2.44⎭	5⎭	2.41	6*b*	2.39	2
2.32⎫	2⎫	2.27	8*b*	2.27	5*b*
2.23⎭	1⎭	—	—	2.21	1
2.16⎫	3⎫	—	—	—	—
2.11⎭	3⎭	2.13	3*b*	2.13	1.5
2.06⎫	1⎫	2.07	6*b*	2.07	2
2.02⎭	1⎭	1.971⎫	3⎫	1.996	1
1.907	1.5	1.881⎭	3⎭	1.881	1*b*
1.804⎫	1.5⎫	1.821	1	1.817	1*b*
1.774⎭	1.5⎭	1.766	1	—	—
1.732	2	—	—	1.728	1
1.695⎫	3⎫	1.704	3*b*	1.697	2
1.675⎭	3⎭	1.672	<1	1.669	1
1.634⎫	1⎫	1.637	<1*b*	1.629	1*b*
1.608⎭	1⎭	1.598⎫	6⎫	1.598⎫	2⎫
1.567⎫	2⎫	1.584⎭	6⎭	1.578⎭	2⎭
1.550⎭	3⎭	1.554	2*b*	1.549	3
1.517	3	1.522	2*n*	1.519⎫	4*n*⎫
1.491	1	1.507	3	1.503⎭	1.5⎭
1.471	1	1.471	<1	1.467	1.5*b*
1.413	1	1.414	3*b*	1.414	1.5*b*
1.380	1*b*	1.374	1*b*	—	—
1.318⎫	1⎫	1.354	1	1.349	1*b*
1.293⎭	1.5⎭	1.339	1	1.312	2
		1.301	4*b*	1.298	3
		1.287	1	1.262	1.5
		1.005	1*b*		

* Intensities were estimated visually with a calibrated intensity strip.
b = broad line.
n = narrow line.
} = band, *d* values for measurements at each edge of band.

patterns of four fibrous sepiolites from Ampandrandava (U.S.N.M. 106827), Tsing Shi-Ling, Inner Mongolia (U.S.N.M. 90719), Chester Co., Pa. (U.S.N.M. 85890), and Little Cottonwood Canyon, Utah (U.S.N.M. C-3808).

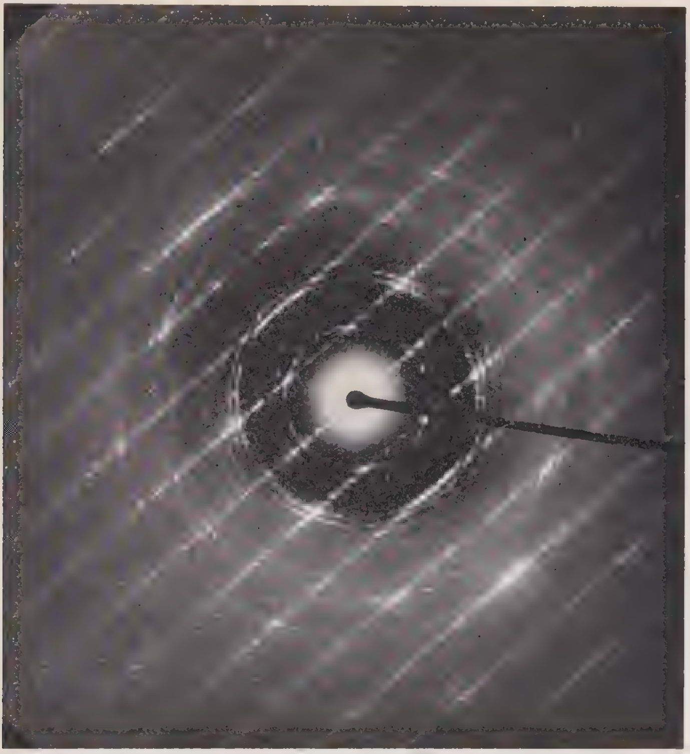

FIG. 3. Electron diffraction fiber diagram of sepiolite from Ampandrandava, Madagascar.

ELECTRON MICROSCOPE AND ELECTRON DIFFRACTION STUDIES

Electron micrographs of untreated loughlinite, loughlinite (leached 6 hrs., Fig. 2), loughlinite (leached 58 days), and sepiolite from Ampandrandava, Madagascar, show that the habits are identical. The width of the individual fibers of loughlinite appear to be about 70 Å.

Electron diffraction powder patterns of loughlinite (leached 6 hrs.) and of sepiolite show small differences similar to those found in the *x*-ray powder patterns. Electron diffraction fiber diagrams, obtained by orienting a very small bundle of fibers with the fiber direction approximately normal to the electron beam, show that the dimension along the fiber direction (c) is 5.26±0.01 Å. in both minerals. Fig. 3 shows such a fiber diagram obtained from a bundle of sepiolite fibers. The rings in this figure are given by those fibers which are randomly oriented.

DISCUSSION

The molecular ratios of loughlinite (leached 6 hrs., analysis A′, Table 1) indicate an ideal formula of $Na_2O \cdot 3MgO \cdot 6SiO_2 \cdot 8H_2O$. The ideal formula for sepiolite, as indicated by the molecular ratios given in Table 1, and as suggested by Caillére (1933), Schaller (1936), and Kauffman (1943), is $4MgO \cdot 6SiO_2 \cdot 8H_2O$. The molecular ratios for the material remaining after leaching loughlinite for 58 days (analysis B, Table 1) appears to be explained by assuming that silica is slowly leached, along with sodium. The resultant leached product has the apparent formula of $3MgO \cdot 5SiO_2 \cdot 8H_2O$.

The experimental work done indicates that loughlinite can be altered to sepiolite (or to a substance closely resembling sepiolite) by two different routes. A comparison of analyses A′ and B (Table 1) indicates that sodium and silica is removed from the loughlinite structure by prolonged leaching, *i.e.*,

$$\underset{\text{(loughlinite)}}{Na_2O \cdot 3MgO \cdot 6SiO_2 \cdot 8H_2O} \xrightarrow[\text{leaching}]{\text{prolonged}} \underset{\text{(sepiolite-like substance)}}{3MgO \cdot 5SiO_2 \cdot 8H_2O + Na_2SiO_3}$$

The alteration of loughlinite to sepiolite or to a sepiolite-like substance on immersion in magnesium salt solutions suggests a simple substitution of 2Na for Mg, *i.e.*,

$$\underset{\text{(loughlinite)}}{Na_2O \cdot 3MgO \cdot 6SiO_2 \cdot 8H_2O} \xrightarrow{Mg^{++}} \underset{\text{(sepiolite-like substance)}}{4MgO \cdot 6SiO_2 \cdot 8H_2O + 2Na^+}$$

It is difficult to imagine a crystal structure which might accommodate transformations of the above nature; thus until further information is obtained the relationship of sepiolite and loughlinite will remain in question.

ACKNOWLEDGMENTS

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COMPILATION OF CHEMICAL ANALYSES AND PHYSICAL CONSTANTS OF NATURAL CORDIERITES

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ABSTRACT

A brief review of previous attempts to correlate the composition and optical properties of cordierite is given, followed by a compilation, including four new analyses, of 62 chemical analyses of natural cordierites together with their refractive indices, optic angles and densities.

INTRODUCTION

The relationship between the chemical composition of cordierite and its optical properties and density has been considered by a number of workers, particularly Oppenheimer (1914, p. 257), Pehrman (1932, p. 1), Shibata (1936, p. 205), Winchell (1937, p. 1175), Thiele (1940, p. 64), Folinsbee (1941, p. 485) and Iiyama (1956, p. 372).

The first notable plot relating α, β and γ, 2V, D and

$$\frac{FeO + 2Fe_2O_3}{FeO + 2Fe_2O_3 + MgO}$$

was drawn by Shibata (1936, p. 223).* Many subsequent diagrams have essentially been modifications of Shibata's diagram.

Early attempts at correlation were frustrated by two main difficulties, one of which is still a considerable drawback. The first problem was the quite unpredictable variation of 2V, which provoked much comment. Thus Brammall & Rao (1936, p. 257) showed that cordierite from the Dartmoor Granite varied in 2Vα from 56°–72° while Tsuboi (1936, p. 337) found that the cordierite in a single rock had a 2V variation from positive near 0° to negative near 0° with most grains having a 2V near 90°. Although most cordierites are negative, a number of well established optically positive cordierites are known (Stewart, 1942, p. 262; Heald, 1950, p. 66; Tilley, 1940, p. 340; Pehrman 1932, p. 5; Mahadevan and Sastry, 1948, p. 363). The second difficulty is the relatively limited range of Fe/Mg variation in common cordierites. Until Shibata (1936, p. 209 and 217) had two mixtures of quartz and iron rich cordierite analyzed there was no information about cordierite richer than 50% of the Fe end member.† Both these analyzed mixtures contained more than 50%

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* Shibata actually used the

$$\frac{FeO + 2Fe_2O_3 + Na_2O}{FeO + 2Fe_2O_3 + Na_2O + MgO}$$

ratio but the Na_2O is usually negligible.

† Read (1929, p. 550) calculated the composition of an Fe rich cordierite from an

quartz and the density and chemical composition had to be calculated from planimetric estimations of the volume of quartz in the cordierite-quartz mixture. Even now, over 20 years later, there is only one more analysis available of an Fe rich cordierite, that by Sambonsugi, quoted by Iiyama (1956, p. 374) and Miyashiro (1957, p. 47). Thus with the exception of these three cordierites, all from Japanese pegmatites, chemical and optical determinations of cordierite cover only the range of 10–54% Fe cordierite, at present.

Winchell (1937, p. 1176) replotted Shibata's data after replacing Na_2O by MnO in the chemical ratio, which then became the same as Niggli's *mg* ratio, $MgO/MgO+FeO+MnO+2Fe_2O_3$ and will be referred to as such below. Winchell's diagram was of uncertain value because "the lack of correlation between chemical composition and optic properties is illustrated by the fact that there are almost as many (seven) samples of cordierite which have been analysed and measured optically that cannot be used in the diagram as the number (eight) used in preparing it" (Winchell, 1937, p. 1178). Neither Winchell nor Shibata considered Pehrman's (1932) data but both showed that despite a poor correlation of refractive indices with Fe/Mg the density increased as the Fe/Mg ratio increased and that when the density was plotted against the refringence a very good correlation was obtained.

In 1940 Thiele (p. 80) made a notable contribution to the data by supplying seven new analyses and he considered that the effect of increase in water content and of substitution of Si and Mg for Al was to decrease the refractive indices and increase the optic angle. Folinsbee (1941, p. 498) rejected these suggestions and proposed that the 2V was related to the alkali content on the grounds that the structures of beryl and cordierite are very similar and it is known that the alkali content has an appreciable effect on the optical properties of beryl. Folinsbee plotted 2V against "the effective alkali content" (Na_2O+K_2O-CaO wt.%) and obtained a smooth curve on the basis of six points. As Heald (1950, p. 67) and Subramaniam (1956, p. 346) have pointed out, many of the analyses published since 1941 do not plot anywhere near to this curve, nor do all the pre-1941 data.

In 1942 Winchell (p. 346) slightly modified his earlier plot of β and D against Fe/Mg while the current edition of Winchell (1951, p. 471) contains a re-modified plot of density, refractive indices and Fe/Mg. Tröger (1952, p. 52) has reproduced a graph nearly identical with the

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analysis of a cordierite-plagioclase mixture but he did not give the refractive indices or density. Because of its unusually Fe rich composition a re-examination of this cordierite is needed.

original Winchell-Shibata diagram but he does not give the evidence upon which his plot is based.

Since Folinsbee's work (*ibid*) little further discussion occurred until lately. Miyashiro in a series of very important papers (principally 1955 and 1957) has concluded (1957, p. 60) that there are at least six polymorphs of cordierite compositions. The different structural states are dominantly controlled by different thermal histories and they show gradations from one to the other, having different order⇄disorder states (as do the alkali felspars). Most of these different structural states are poorly understood. However, "most cordierites, if not all, in ordinary metamorphic rocks, pegmatites and quartz veins belong to the low subdistortional class, pseudohexagonal and unstable at all temperatures" (Miyashiro, 1957, p. 60.) However, this cordierite, known as low cordierite, has a considerable degree of possible structural order⇄disorder variation and clearly this affects the refractive indices slightly and the optic angle very considerably. Thus it is not surprising that the correlation of composition with refringence and 2V has not been very successful and Iiyama (1956, p. 380) and Miyashiro (1957, p. 48) have shown that 2V is largely but not entirely controlled by the "distortion index" ($=\Delta$) which is obtained from an x-ray diffractogram while β is lower for a given Fe/Mg ratio the higher the distortion index. This index, which is zero for hexagonal indialite and roughly indicates a measure of deviation from hexagonal symmetry, decreases with increase of disorder and of temperature of crystallization but Iiyama (1956, p. 372) and Miyashiro (1957, p. 53) have suggested that cordierites from volcanic rocks have lower refractive indices than cordierites from other natural environments because they form a separate group of cordierites known as high (temperature) cordierite. At present, however, there are only two chemical and optical analyses of volcanic cordierites available and the accuracy of one of these (No. 57, Table 1) is uncertain.*

Heating low temperature cordierite lowers its refractive indices and Iiyama (1956, p. 387) has recommended that the most accurate determination of Fe/Mg without chemical or x-ray analysis is to be obtained by heating a given cordierite for 10 mins. at 1000° C. cooling and then determining the refractive indices and obtaining Fe/Mg from a graph which has been drawn from similarly treated and chemically analyzed natural cordierites. The heating is intended to ensure that the cordierite sample is in a similar structural state (*i.e.* having a similar distortion index) to the cordierites used in preparing the graph. At present this plot is based on the data of 8 cordierites.

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* Shand, 1943, *Am. Mineral.*, **28**, p. 393 gives an analysis of an iron-free cordierite (which has 1.24% CaO) from the wall of a glass furnace tank and this high temperature form has very low refractive indices, $\alpha=1.5223$, $\gamma=1.5270$, $2V\alpha=82\frac{1}{2}°$, $D=2.520$ at 20° C.

Iiyama (1956, p. 387) has pointed out that the approximate composition of a cordierite can also be obtained by measurement of $d\theta_{004}$. He suggests that if $2\theta_{004}$ can be measured to ±0.02° then $Fe^2+Mn/Fe^2+Mn+Mg$ can be estimated to ±10% and if the refractive indices can be measured to ±0.003 and the sample has previously been given the standard heat treatment then the ratio can be estimated to ±5–8%, whereas the accuracy when the refractive indices of unheated cordierite are determined "depends greatly on that of the estimated Δ value." There is, however, no published plot, or list, of all the available data on natural cordierite and so it is difficult to know with what accuracy Fe/Mg can be estimated from unheated samples. In the light of current developments in our understanding of cordierite it is now certain that all really precise cordierite studies must include *x*-ray analysis. Nevertheless much could be achieved if all the available information on the optics and density of cordierites was fully utilized and so this paper gives a collection of data on natural cordierite (Table 1 and Figs. 1 to 6) including four unpublished analyses (Nos. 32, 50, 55 and 59).

No compilation of the published data is or has been available. Oppenheimer (1914, p. 258) gave a comprehensive list of references to the early literature dealing with cordierite optics and chemistry and he tabulated most of the refractive indices then determined, but only about half a dozen of the chemical analyses are given. Winchell's (1938, p. 1179) list of references is the most comprehensive available at present and Folinsbee (1941, p. 493) supplements this but Winchell did not reproduce the original data in his paper, which is in any case, 20 years old now.

Discussion

The enigmatic role of water in cordierite has been recognized for some time (Gossner and Reindl, 1932, p. 330; Winchell, 1937, p. 1179; Thiele 1940, p. 84; Tilley, 1940, p. 341). Recently Iiyama (1958, p. 796), on the basis of thermobalance experiments on the Laramie Range cordierite No. 2 has claimed that the decrease in refractive indices and increase in 2V which occurs when low temperature cordierite is heated and transformed to high temperature cordierite is related to loss of water, the transformation taking place at about 525° C. at atmospheric pressure but at 750° C. when the water vapor pressure is about 600 bars.* An approximate estimate of the correlation between water content and refractive index in natural cordierites was obtained from the data in Table 1 thus. If

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* Cf. Thiele, 1940, p. 84 who concluded that increase in water content decreased refringence.

TABLE 1. CHEMICAL ANALYSES, SPECIFIC GRAVITIES AND REFRACTIVE INDICES OF 62 CORDIERITES

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21
SiO_2	49.46	47.09	50.15	49.54	49.18	48.52	48.05	49.17	50.65	49.12	47.96	46.08	49.05	49.60	48.11	48.04	49.47	47.43	48.33	49.50	46.75
Al_2O_3	33.58	34.86	33.07	33.42	32.28	35.25	33.37	33.10	30.26	32.25	31.52	32.85	33.08	31.19	33.02	33.41	33.18	33.34	32.31	33.01	33.64
TiO_2	0.01	0.03	0.38	0.03	n.d.	tr.	n.d.	n.d.	n.d.	n.d.	0.00	0.05	n.d.	n.d.	tr.	0.06	n.d.	0.03	n.d.	n.d.	0.00
Fe_2O_3	0.14	0.11	1.52	0.14	0.08	0.41	0.09	n.d.	n.d.	n.d.	1.03	0.12	0.83	1.19	0.81	0.01	1.15	0.07	2.22	0.38	0.72
FeO	2.12	2.59	2.22	3.75	4.05	3.43	4.01	4.34	4.10	5.09	3.24	4.08	4.38	4.10	4.49	5.04	4.53	4.91	3.74	5.12	4.89
MgO	12.06	11.92	11.01	11.34	11.25	10.40	11.11	11.45	11.09	11.96	12.16	9.56	11.04	11.13	10.95	10.32	10.80	9.70	10.66	10.42	10.43
CaO	0.03	0.04	0.29	0.10	n.d.	tr.	0.05	n.d.	n.d.	tr.	0.00	1.32	n.d.	0.23	tr.	0.04	n.d.	0.04	0.84	n.d.	0.04
Na_2O	0.14	0.78	0.14	0.32	0.21	0.13	0.26	n.d.	n.d.	n.d.	0.33	0.62	n.d.	0.21	0.12	1.12	0.17	0.61	n.d.	n.d.	0.24
K_2O	0.30	tr.	0.08	0.01	0.44	0.16	0.39	n.d.	n.d.	n.d.	tr.	0.49	n.d.	0.06	tr.	0.30	0.04	tr.	n.d.	n.d.	0.56
MnO	0.08	0.05	0.12	0.09	n.d.	0.08	tr.	0.04	0.60	tr.	1.09	0.21	n.d.	n.d.	0.07	0.20	n.d.	0.12	n.d.	0.29	0.31
H_2O^-	1.71	2.21	1.37	1.32	2.04	2.06	2.75	1.20	2.66	0.32	2.80	4.88	1.64	2.33	2.72	1.81	1.16	2.74	1.44	1.62	2.63
H_2O^-	0.10	0.65	0.09	0.03	0.27	0.12								0.08	0.11	0.23	0.04	1.01			0.10
	99.73	100.33	100.44	100.10	99.80	100.57	100.08	99.30	100.00	98.74	100.13	100.26	100.02	"100.22"	"100.16"	100.58	100.54	100.00	99.54	100.34	100.33
Si	4.99	4.80	5.02	4.98	5.02	4.91	4.93	4.98	5.21	4.99	4.93	4.87	4.97	5.07	4.93	4.91	4.97	4.92	4.94	5.02	4.81
Al	1.01	1.20	0.98	1.02	0.98	1.09	1.07	1.02	0.79	1.01	1.07	1.13	1.03	0.93	1.07	1.09	1.03	1.08	1.06	0.98	1.19
Al	2.98	2.99	2.92	2.94	2.91	3.07	2.96	2.94	2.87	2.85	2.75	2.96	2.92	2.83	2.91	2.93	2.90	3.00	2.83	2.96	2.93
Ti	0.00	0.00	0.03	0.00		0.00					0.00	0.01			0.00	0.00		0.00			0.00
Fe^3	0.01	0.01	0.11	0.01	0.01	0.03	0.01				0.08	0.01	0.06	0.09	0.06	0.00	0.08	0.01	0.17	0.03	0.05
Fe^2	0.18	0.22	0.19	0.32	0.34	0.29	0.34	0.37	0.35	0.43	0.28	0.35	0.37	0.35	0.38	0.43	0.38	0.43	0.32	0.43	0.42
Mn	0.01	0.00	0.01	0.01		0.01	0.00	0.00	0.05	0.00	0.10	0.02			0.01	0.02		0.01		0.02	0.03
Mg	1.81	1.80	1.64	1.70	1.71	1.57	1.67	1.73	1.70	1.81	1.86	1.51	1.67	1.69	1.68	1.57	1.62	1.50	1.62	1.57	1.60
Ca	0.00	0.01	0.03	0.01		0.00	0.01			0.00	0.00	0.15		0.02	0.00	0.00		0.00	0.09		0.00
Na	0.02	0.16	0.03	0.06	0.04	0.01	0.05				0.07	0.13		0.04	0.02	0.22	0.04	0.12			0.05
K	0.04	0.00	0.01	0.00	0.06	0.01	0.05				0.00	0.06		0.01	0.00	0.04	0.01	0.00			0.07
$\frac{Fe^2+Mn\times100}{Fe^2+Mn+Mg}$	9.1	11.0	10.9	15.9	16.7	15.8	16.9	17.5	19.1	19.3	17.0	20.2	18.2	17.2	18.8	22.2	19.0	22.6	16.5	22.3	21.9
D	2.583– 2.603		2.588	2.591	2.581		2.581	2.60	2.60	2.59			2.593	2.598	2.589		2.581		2.71	2.607	
mg	0.906	0.889	0.840	0.838	0.830	0.830	0.829	0.825	0.809	0.807	0.805	0.795	0.794	0.793	0.788	0.778	0.777	0.771	0.767	0.765	0.761
α	1.530	1.534	1.527	1.539	1.536	1.534	1.536				1.534	1.541	1.539	1.541	1.542	1.539	1.533	1.538		1.540	1.542
β	1.535	1.539	1.532	1.544	1.541	1.536							1.544	1.545		1.544	1.537	1.542		1.546	1.546
γ	1.538	1.543	1.538	1.546	1.543	1.545	1.543				1.543	1.550	1.547	1.547	1.548	1.547	1.541	1.554		1.548	1.551
$2V\alpha$	84½	77	92	66	69½	89	65½				104	71½	70	78¼	74	68	80	72	106	65	68

	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42
SiO_2	49.14	48.58	48.65	48.44	48.32	49.51	50.13	54.63	49.15	48.61	49.58	48.17	48.77	46.99	48.85	48.37	48.15	47.33	48.43	47.69	48.19
Al_2O_3	32.84	31.47	32.58	31.74	35.02	32.46	32.11	29.32	34.60	31.97	32.71	32.53	31.01	32.72	32.52	29.22	30.90	32.59	32.36	32.52	33.45
TiO_2	n.d.	n.d.	n.d.	tr.	n.d.	0.00	n.d.	0.00	n.d.	n.d.	0.00	n.d.	0.07	0.10	n.d.	n.d.	tr.	0.02	n.d.	tr.	0.01
Fe_2O_3	0.63	1.85	1.75	1.29	n.d.	0.29	2.24	1.13	1.40	1.45	0.00	1.21	2.83	0.16	2.94	2.20	0.75	0.15	n.d.	0.63	0.55
FeO	5.04	4.90	4.79	4.63	5.76	6.51	6.37	5.15	4.25	7.10	7.26	6.63	5.19	7.12	5.28	7.07	7.75	8.49	8.55	8.04	8.40
MgO	10.40	10.68	10.02	9.39	8.83	9.60	8.47	7.82	7.68	9.95	8.65	8.86	8.91	8.38	8.50	9.54	8.50	8.07	7.81	7.56	7.95
CaO	n.d.	tr.	n.d.	0.70	0.08	0.00	n.d.	0.00	0.55	0.44	0.00	tr.	0.43	0.06	0.43	1.92	0.35	0.09	0.46	0.52	0.17
Na_2O	n.d.	0.35	0.15	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.24	1.39	0.16	0.85	n.d.	0.40	0.53	n.d.	n.d.	0.53	0.22
K_2O	n.d.	n.d.	0.04	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.09	0.50	0.06	0.30	n.d.	n.d.	tr.	0.48	n.d.	0.42	0.02
MnO	0.19	n.d.	n.d.	0.27	0.12	0.10	0.32	0.02	0.94	tr.	0.00	n d.	0.31	0.42	0.28	0.42	0.06	0.06	n.d.	0.04	0.18
H_2O^+	1.84	1.82	2.39	3.32	1.73	1.07	0.99	0.82	2.15	1.12	1.31	1.12	1.99	3.07	1.82	1.84	3.35	1.88	1.55	1.85	0.67
H_2O^-		0.14	0.10	0.20	0.30							0.10	0.29	0.05				0.20	1.32	0.55	0.01
	100.08	99.79	"100.26"	99.98	100.16	99.54	100.63	99.89	100.72	100.64	99.84	100.51	100.02	100.22	100.62	100.58	100.21	99.94	100.48	100.35	99.82
Si	5.00	4.99	4.97	5.03	4.92	5.04	5.08	5.52	4.99	4.95	5.07	4.94	5.04	4.92	5.00	5.02	5.04	4.94	5.03	4.99	4.95
Al	1.00	1.01	1.03	0.97	1.08	0.96	0.92	0.48	1.01	1.05	0.93	1.06	0.96	1.08	1.00	0.98	0.96	1.06	0.97	1.01	1.05
Al	2.93	2.80	2.89	2.82	3.13	2.93	2.91	3.00	3.13	2.79	3.01	2.87	2.81	2.95	2.91	2.60	2.86	2.95	2.99	3.00	3.00
Ti				0.00		0.00		0.00			0.00		0.01	0.01			0.00	0.00		0.00	0.00
Fe³	0.05	0.14	0.13	0.10		0.02	0.17	0.08	0.10	0.11	0.00	0.09	0.22	0.01	0.22	0.17	0.06	0.01		0.05	0.04
Fe²	0.43	0.42	0.41	0.40	0.49	0.55	0.54	0.44	0.36	0.61	0.62	0.57	0.44	0.62	0.45	0.61	0.68	0.74	0.74	0.70	0.72
Mn	0.02			0.02	0.01	0.01	0.02	0.00	0.08	0.00	0.00		0.02	0.04	0.02	0.04	0.01	0.01		0.00	0.02
Mg	1.58	1.64	1.52	1.46	1.33	1.46	1.28	1.18	1.16	1.51	1.31	1.35	1.37	1.31	1.30	1.48	1.33	1.25	1.21	1.18	1.21
Ca		0.00		0.08	0.01	0.00		0.00	0.06	0.05	0.00	0.00	0.05	0.01	0.04	0.21	0.04	0.01	0.06	0.06	0.02
Na		0.07	0.03								0.05	0.28	0.03	0.17			0.08	0.11		0.05	0.04
K			0.01								0.01	0.06	0.01	0.04			0.00	0.06		0.03	0.00
$\frac{Fe^{2}+Mn\times100}{Fe^{2}+Mn+Mg}$	22.0	20.4	21.3	22.7	27.3	27.8	30.7	27.0	27.5	28.7	31.9	29.5	25.6	33.6	27.0	30.4	34.0	37.5	38.0	37.2	37.7
D	2.610	2.603	2.614	2.632	2.603	2.588	2.696		2.615	2.60		2.660	2.629		2.620	2.598		2.63	2.642	2.64	2.631
mg	0.760	0.744	0.736	0.735	0.727	0.713	0.698	0.694	0.680	0.680	0.680	0.674	0.666	0.660	0.650	0.644	0.641	0.623	0.620	0.609	0.607
α		1.540	1.541	1.536		1.537		1.540		1.530	1.538	1.552	1.546	1.551			1.537	1.543	1.548	1.538	1.544
β		1.546	1.547	1.543		1.543		1.545		1.534	1.544	1.560		1.560				1.549	1.555	1.542	1.550
γ		1.549	1.550	1.546		1.548		1.552		1.537	1.547	1.561	1.558	1.562			1.546	1.555	1.559	1.547	1.556
2Vα		65½	66½	68½		81½		77		81	75-81 av. 78	43½		42		85-99		100	53	96	85-95

(Continued on next page)

TABLE 1 (*continued*)

	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62
SiO_2	40.92	48.88	48.43	48.6	48.09	48.40	48.18	47.18	49.14	49.50	50.09	47.88	47.32	49.62	48.58	49.74	46.69	44.64	43.55	43.27
Al_2O_3	31.06	33.07	32.36	30.5	30.87	32.15	33.86	32.10	30.99	32.45	31.78	32.40	33.36	28.72	32.44	35.21	32.00	29.96	30.68	30.25
TiO_2	n.d.	n.d.	n.d.	n.d.	n.d.	0.05	n.d.	0.16	tr.	0.06	0.00	0.00	0.00	n.d.	n.d.	n.d.	0.34	n.d.	tr.	tr.
Fe_2O_3	n.d.	0.53	n.d.	n.d.	2.63	0.00	0.34	0.28	2.49	0.40	0.78	0.64	0.96	n.d.	3.15	5.65	0.39	2.72	1.50	1.08
FeO	7.99	8.05	8.55	10.7	7.08	9.80	9.34	9.26	7.03	8.90	8.71	9.65	9.23	11.58	9.17	3.00	12.04	11.02	13.17	15.31
MgO	6.71	7.04	7.81	8.2	7.82	7.25	7.11	6.99	7.38	6.73	6.69	6.94	6.89	8.64	6.63	4.30	5.91	3.08	2.70	1.48
CaO	tr.	0.04	0.46	n.d.	0.60	tr.	n.d.	0.16	0.28	0.57	0.00	0.08	0.04	0.23	n.d.	1.05	0.18	0.02	0.16	0.10
Na_2O	0.72	0.09	n.d.	n.d.	n.d.	0.39	n.d.	0.65	0.10	0.46	0.00	0.23	0.43	n.d.	n.d.	n.d.	0.28	2.05	1.32	3.53
K_2O	8.60	n.d.	n.d.	n.d.	n.d.	0.07	n.d.	0.59	n.d.	0.39	0.07	0.07	0.19	n.d.	n.d.	n.d.	0.16	0.75	0.32	0.90
MnO	n.d.	0.06	1.32	0.1	1.22	0.23	0.11	0.19	1.01	0.11	0.00	0.10	0.26	1.51	tr.	n.d.	0.09	1.86	2.88	1.20
H_2O^+	3.22	2.21	1.55	1.5	2.31	1.58	1.30	1.97	1.69	0.62	1.43	1.92	1.42		n.d.	1.74	1.95	3.85	3.72	2.88
H_2O^-		0.20				0.16			n.d.	0.00		0.24	tr.							
	99.22	100.17	100.48	99.8	100.62	100.11	100.24	99.53	100.11	100.19	99.55	100.15	100.40	100.30	99.97	100.69	100.03	99.95	100.00	100.00
Si	4.62	5.05	5.03	5.08	5.00	5.03	4.95	4.96	5.09	5.07	5.17	5.00	4.90	5.14	4.98	5.03	4.93	4.93	4.83	4.83
Al	1.38	0.95	0.97	0.92	1.00	0.97	1.05	1.04	0.91	0.93	0.83	1.00	1.10	0.86	1.02	0.97	1.07	1.07	1.17	1.17
Al	2.75	3.07	2.99	2.83	2.78	2.97	3.05	2.94	2.87	2.99	3.03	2.98	2.97	2.64	2.90	3.23	2.92	2.82	2.84	2.81
Ti						0.00		0.01	0.00	0.00	0.00	0.00	0.00				0.03		0.00	0.00
Fe^3		0.04			0.21	0.00	0.03	0.02	0.19	0.03	0.06	0.05	0.07		0.24	0.43	0.03	0.23	0.13	0.27
Fe^2	0.75	0.70	0.74	0.93	0.62	0.85	0.80	0.81	0.61	0.76	0.75	0.84	0.80	1.00	0.79	0.25	1.06	1.02	1.22	1.43
Mn		0.01		0.01	0.11	0.02	0.01	0.01	0.09	0.01	0.00	0.01	0.03	0.13	0.00		0.01	0.17	0.27	0.11
Mg	1.13	1.08	1.21	1.27	1.21	1.12	1.09	1.10	1.14	1.03	1.03	1.08	1.07	1.33	1.01	0.65	0.93	0.50	0.45	0.25
Ca	0.00	0.01	0.05		0.07	0.00		0.04	0.03	0.06	0.00	0.01	0.01	0.02		0.10	0.02	0.00	0.02	0.01
Na	0.16	0.02				0.08		0.13	0.02	0.09	0.00	0.05	0.09				0.05	0.44	0.28	0.76
K	1.23					0.01		0.08		0.05	0.01	0.01	0.02				0.02	0.11	0.05	0.13
$\frac{Fe^2+Mn\times100}{Fe^2+Mn+Mg}$	40.1	39.4	41.4	42.5	37.4	43.6	42.9	43.1	38.0	42.6	42.1	44.1	43.6	46.0	43.9	27.8	53.5	70.3	76.7	86.1
D	2.77	2.647	2.642	2.64	2.636	2.66	2.623		2.651	2.622	2.650			2.65	2.627	?2.64			2.72	2.75
mg	0.599	0.594	0.586	0.575	0.566	0.564	0.564	0.562	0.561	0.560	0.559	0.546	0.543	0.540	0.494	0.488	0.458	0.263	0.216	0.128
α		1.549				1.543	1.536	1.543	1.550	1.543	1.543	1.547	1.544				1.548	1.560	1.555	1.558
β						1.553	1.539	1.549		1.550	1.548	1.552	1.550		1.541		1.554	1.574	1.566	1.568
γ		1.560				1.555	1.543	1.552	1.563	1.557	1.553	1.557	1.555				1.558	1.578	1.571	1.573
$2V\alpha$		74½				71¾	100	74–81 av. 79		81	91½	77 86	76		86		72–78 av. 76	55	66	68½

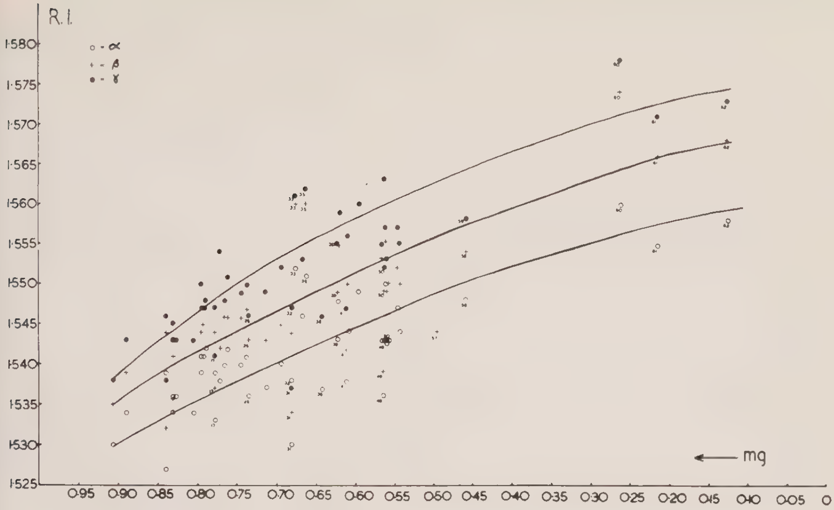

FIG. 1. Plot of refractive indices of natural cordierites against Niggli *mg* ratio.

cordierites rich in water have higher refringences than those with lower water contents but similar *mg* ratios then the hydrous cordierites ought to lie above the curves in Fig. 1 and the lower water-bearing cordierites ought to lie below. The average water content (1.94%) of 36 analyzed cordierites, with *mg* between 0.9 and 0.5, accompanied by refractive indices was obtained (No. 31 was excluded). The differences, positive or negative, from this average, of each of the water contents of the 36 cordierites was calculated. The mean refractive index

$$= \left(\frac{\alpha+\beta+\gamma}{3} \quad \text{or} \quad \frac{\alpha+\gamma}{2}\right)$$

of each of the cordierites was obtained and the difference between this value and the mean value

$$\left(\frac{\alpha+\beta+\gamma}{3}\right)$$

obtained from the curves for α, β and γ in Fig. 1, using the known *mg* value was calculated. The resulting correlation coefficient between the water differences and the refractive index differences was equal to $+0.37$, which is not a good correlation coefficient but since Δ has not been considered, may be significant, suggesting that increase in water content tends to increase refractive index. Chambers (1952, p. 62) gives $\gamma = 0.325$ for 37 observations when $P = 0.05$.

In view of the possibility that volcanic cordierites have lower refractive indices than other natural cordierites, in both Figs. 1 and 2* the numbers of samples with particularly low refractive indices have been inserted. It is notable that in addition to the volcanic cordierites No. 31 from Kasyo-to, Formosa and No. 57 from Cabo de Gata, Spain, Nos. 3, 41 and 49 also have particularly low refractive indices for their *mg* values although No. 3 from an anthophyllite gneiss, Attu, Finland, No. 41 from a hornfels at Belhelvie, Scotland and No. 49 from a cordierite-biotite-microcline-andesine pegmatite at Degero, Finland, are unlikely to be

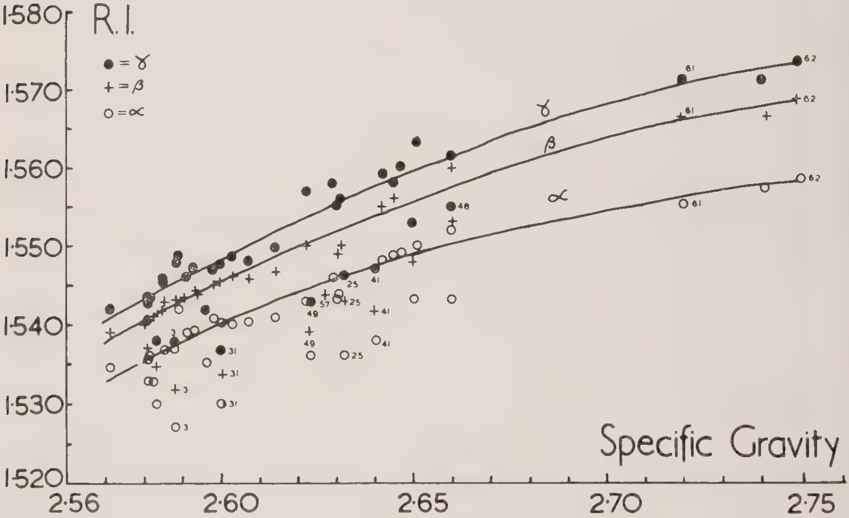

FIG. 2. Plot of specific gravities of natural cordierites against refractive indices.

particularly high temperature cordierites. Although these last three cordierites, with 1.37, 1.85 and 1.30% H_2O respectively, have less than the average water content their deficiency in water is not extreme; many other cordierites have much less water and so water deficiency cannot explain their extremely low refringence. It is possible that these three cordierites are exceptionally ordered and have very high Δ values. It is also of interest that Oppenheimer (1914, p. 299) gives for an unanalyzed cordierite from Cabo de Gata, Spain, $\alpha = 1.5346$, $\beta = 1.5391$, $\gamma = 1.5432$, $2V = 85\frac{1}{2}°$ (presumably negative), $D = 2.571$ which, although β is lower than Osann's (1888. p. 704) determination ($\beta = 1.5438$) on Cabo de Gata cordierite, plots a little above the curves in Fig. 2. Oppenheimer's density is suspiciously low for cordierite. It is important that a reexamination of the Cabo de Gata cordierite be made for Osann's analysis is over 70 years old, its calcium and water contents were never determined and yet it is one of only two volcanic cordierite analyses available.

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* For economy of space the data used in compiling Figs. 2 and 6 which do not appear in Table 1 are not reproduced. Most of the information is contained in Shibata, 1936, p. 420.

Figs. 3 and 4 show great scatter and there is only a very poor correlation of 2V with either *mg* or Na_2O+K_2O-CaO. With the exception of Nos. 19, 28 and 43 which undoubtedly have erroneous density determinations, a good correlation of *mg* and density is shown by Fig. 5, whereas the plots of *mg* versus refringence (Fig. 1) show that little reliability can be attached to the drawn curves. Figure 2 (density versus refringence), however, shows rather less scatter of the points from the drawn curves.

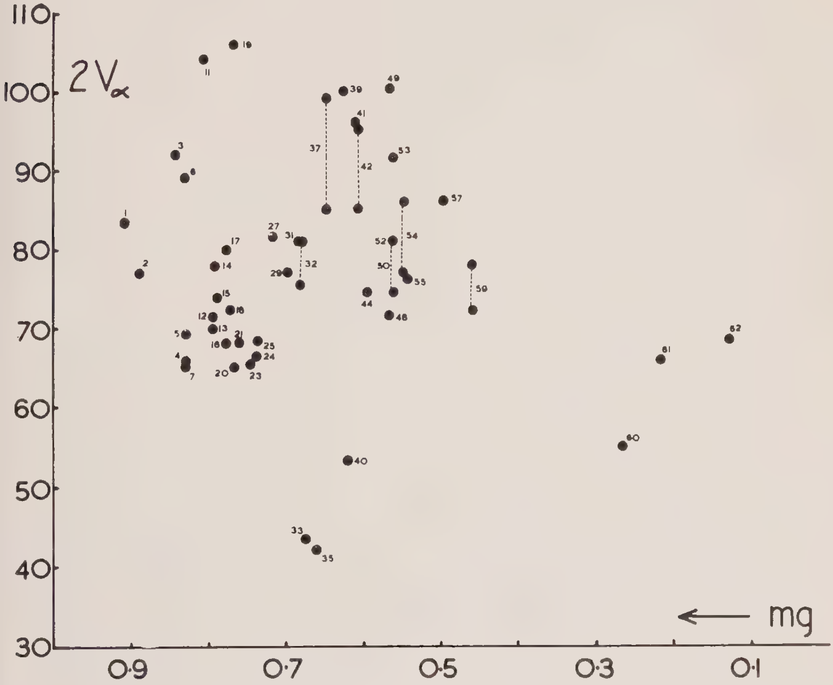

FIG. 3. Plot of 2Vα of natural cordierites against Niggli *mg* ratio.

This apparent better correlation between density and refringence (Fig. 2) compared with *mg* and refringence (Fig. 1) is puzzling. There are several possible explanations, the most obvious one being that density has, on the whole, been determined more accurately than Fe/Mg, though proof of this is difficult. Winchell (1937, p. 1178) suggested that the better correlation of density and refringence resulted from the fact that density could be determined on the same crystal as was used for the refractive index determinations whereas the chemical analysis was an average of all the cordierites in a rock. However, it seems very doubtful whether more than an insignificant proportion of the actual density and refractive index results have been determined on the same grains. Iiyama (1956, p. 389) has indicated that with increase in disorder in cordierite density increases and so does refringence so that some of the better correlation might be the result of this relationship, particularly over the density range 2.58–2.61 (Fig. 2). If density does decrease with increase in Δ then volcanic cordierites, which have high Δ values, ought to plot below the line in Fig. 5 and in fact both No. 30 and No. 53 fall well below the line.

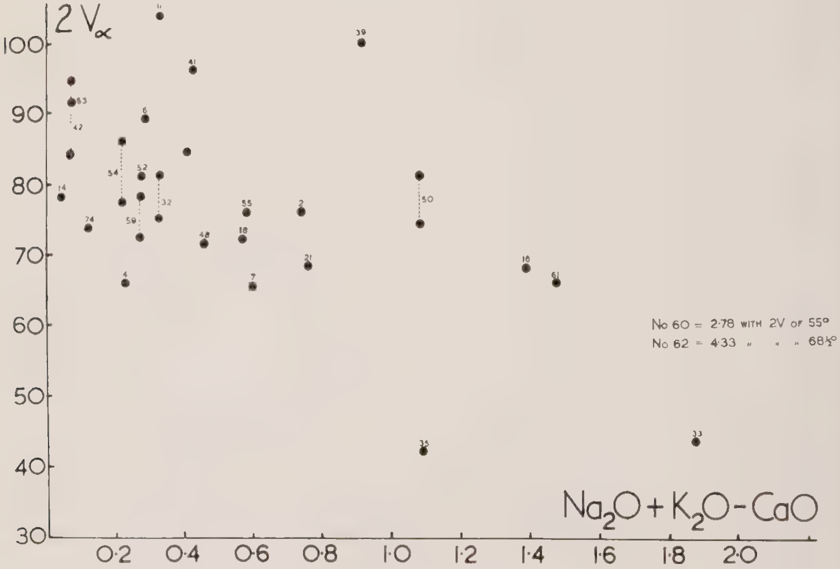

FIG. 4. Plot of $2V\alpha$ of natural cordierites against "effective alkali content" $= Na_2O + K_2O - CaO$ wt. %.

Following a suggestion kindly made by Dr. M. H. Hey a plot of the calculated volume of the unit cell (in cubic Ångstroms) against *mg* was made for 34 cordierites. For constant composition this volume ought to depend only on the degree of order-disorder in each individual cordierite. But the two volcanic cordierites, 30 and 53 do not support the view that there is any difference between the cell volumes of differently

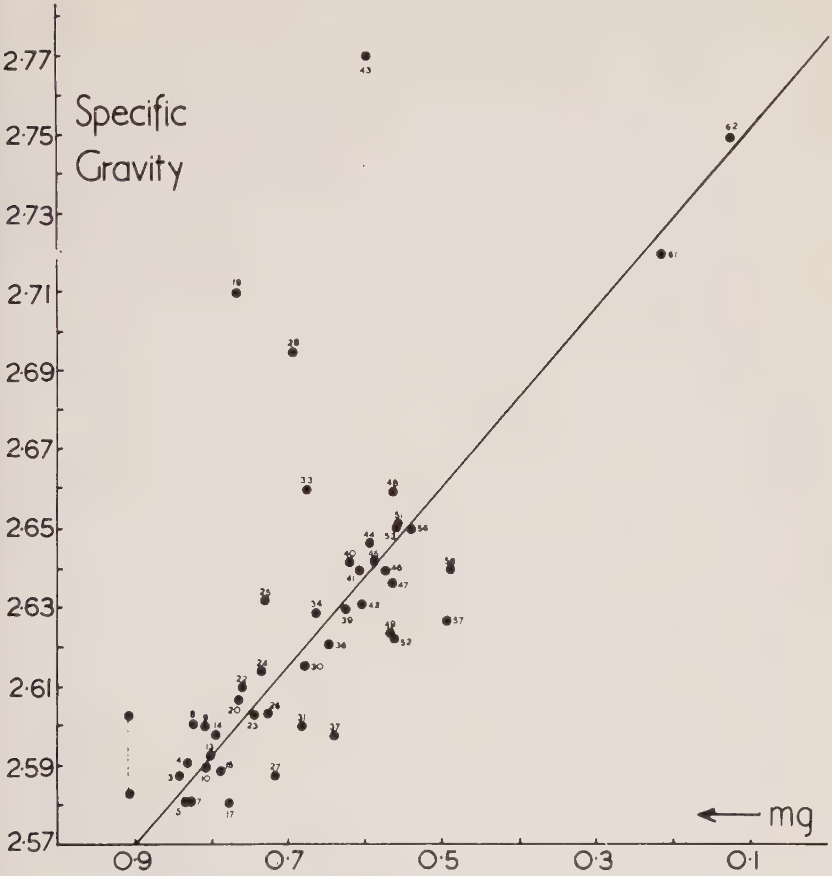

FIG. 5. Plot of specific gravities of natural cordierites against Niggli *mg* ratio.

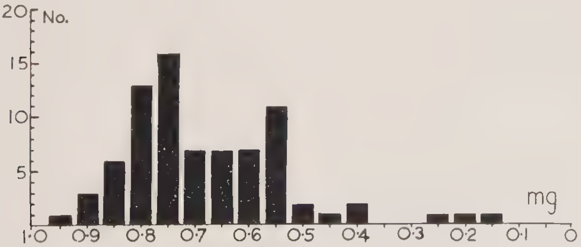

FIG. 6. Histogram to show frequency of occurrence of *mg* values of 79 natural cordierites. Class interval 0.05 *mg*.

ordered cordierites as they plot in the middle of a frequency distribution (whose maximum is about 3.9×10^{22} cubic Å) and the scatter of which seems to be largely the result of scattered density determinations. Such a plot relies unduly on the accuracy of the density determination and cordierites which plot away from the frequency distribution also show excessive scatter in Fig. 5. Dr. M. H. Hey also suggested a correlation of specific refractivity $(n-1)/d$ with *mg* but a plot of 30 cordierites showed that outstanding points did not belong to cordierites with unusual chemistry e.g. H_2O, Na, Ca, Fe or Mg.

Fig. 6 shows that 85% of the analyzed natural cordierites have *mg* values between 0.55 and 0.85, an observation of some value in the calculation of Niggli Katavariants.

Recent discoveries of the various structural states of cordierite suggest that an *x*-ray examination of cordierite from granites, such as the Dartmoor Granite S. W. England, may throw valuable light on the thermal history of the rocks and on the granite problem generally, in a similar manner to that shown by felspars and quartz.

Acknowledgments

I wish to thank Drs. Bernard Evans and G. Chinner for allowing me to include unpublished analyses; Dr. A. Miyashiro of the University of Tokyo, for his kind assistance over certain Japanese references; Dr. F. C. Phillips and Mr. R. Bradshaw for revising the manuscript and Drs. M. H. Hey and W. Schreyer for their helpful interest in the problem. The writer would be grateful to know of omissions and additions to Table 1.

Localities, Sources and Notes to Table 1

All the cell contents given have been calculated to 18 oxygens excluding water. Most of the pre-1900 analyses (whose value is dubious) do not give the state of oxidation of the iron and the writer has assumed, or calculated it to be FeO. There are many discrepancies and misquotations in the early literature, for example compare Dana 3rd ed, 1850, p. 344, George Putnam, London, and Dana, 6th ed., 1892, p. 420, Chapman & Hill, London, but these have not all been catalogued for economy of space. All, or nearly all of the water contents determined before 1900 are lost on ignition, while later determinations are mostly by the Penfield (1894) method which tends to give consistently low results (Riley, 1958).

An empty space indicates that no information is available; n.d. denotes "not determined."

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UNUSUAL ETCH PITS IN QUARTZ CRYSTALS

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Abstract

Synthetic and natural quartz crystals have been etched in 48% HF in a manner described by Arnold[7] and have been found to exhibit deep etch tubes which are ribbon-like in shape. Examples of these etch tubes are shown and possible explanations for their formation are given.

Introduction

Recently there has been interest in the structural defects in crystalline quartz and their effect on its physical properties. Bömmel, Mason and Warner[1] have postulated that dislocations exist in quartz. King[2] has suggested that the mechanical properties of quartz are dependent upon its defect structure and has observed differences between synthetic and natural quartz. Cohen[3,4,5] concludes that amethyst and smoky quartz derive their color from defects associated with impurity atoms. The present work was suggested by the discovery of Arnold[6] that concentrated hydrofluoric acid produced deep etch pits, actually etch tubes, in a synthetic quartz crystal obtained from Clevite Corporation. After the present work was completed, the authors discovered similar work had been carried on by a group at Clevite Corporation.[7] Although their results are similar, they differ enough in detail to warrant presentation of our results.

Etch tubes similar to those described below have been observed in minerals such as barite, dolomite, colemanite, apophyllite and topaz. An excellent summary of these observations has been provided by Honess.[8] Miss L. C. Lovell[9] of these Laboratories has recently observed etch pits in apatite which bear a resemblance to those discussed below but which are smaller in size by three orders of magnitude.

Experimental

Several crystals were etched in 48% HF for 48 hours at room temperature. One crystal (#1) was of natural quartz and was cut such that two basal (0001) planes were parallel, making a plate $\frac{1}{2}$ inch thick. Five of the prism faces, $(10\bar{1}0)$, were neither cut nor polished. The sixth prism face had been trimmed with a diamond saw.

A second crystal (#2) was a synthetic stone grown on a Y-bar seed about 3″ long and $\frac{1}{8}$″ square. (The long sides of a Y-bar seed are (0001) and $(11\bar{2}0)$ planes, the ends are prism faces.) This crystal is similar to the one used by Arnold.

A third crystal (#3) was synthetic also and was grown on a CT plate, that is, the large planes of the seedplate were minor rhombohedral faces, $(01\bar{1}1)$.

Three other CT plate crystals and two Y-plate crystals were also etched but were not polished.

After etching, the crystals were washed in distilled water and dried. Crystals #1, 2 and 3 were then polished on two parallel faces so that etch pits could be observed conveniently. Crystals #1 and 2 were polished on two (0001) faces. Crystal #3 was polished on two $11\bar{2}0$ faces.

OBSERVATIONS

1. *The Natural Crystal*

Figure 1 is a photograph of the natural crystal (#1) after etching and polishing. The etch pits appear as small tubes which penetrate three of the $(10\bar{1}0)$ faces of the crystal to depths greater than one centimeter. A close examination of the tubes revealed no particular pattern in their formation, that is, there was no evidence that they formed in rows as is often observed where etch pits are found to originate because of dislocations. Although the tubes are, on the average, perpendicular to the (1010) faces they are seldom exactly so and individuals are often as much as 10° off the perpendicular.

It should be noted that the crystal shown in Fig. 1 had regions of smokiness visible in it. These darker regions appear near the edge of the crystal near five $(10\bar{1}0)$ faces. Since there are etch tubes in three of these regions but not in the other two there appears to be no relation between smokiness and the formation of this type of etch pit.

After photographing the crystal the regions containing the etch pits were cut from the crystal with a diamond saw and the remaining stone was again etched for 48 hours in 48% HF. No etch tubes were observed on any of the faces.

2. *Y-bar Synthetic Crystal* (*BTL*)

It was noted that in the case of the Y-bar crystal the etch tubes clustered along a line running down the center of the large face which was approximately $(11\bar{2}0)$. These etch tubes all terminate at or near the seed and appear to fan out from the seed in the direction of the $(11\bar{2}0)$ face in the manner of bristles of a worn brush. The etch tubes appearing away from this centrally located cluster all originate at inclusions of impurities which are usually visible under the microscope. Figure 2 shows two inclusions and the etch tubes pointing toward them.

Almost all etch tubes in crystal #2 originate at the $(11\bar{2}0)$ faces. The initial form of the tubes is similar to a normal etch pit much elongated in

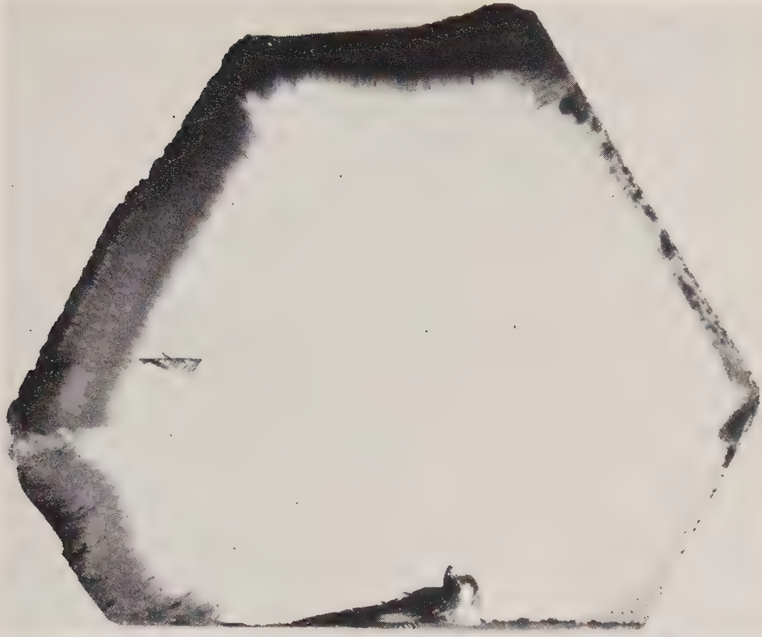

FIG. 1. Etch tubes in natural crystal (2×).

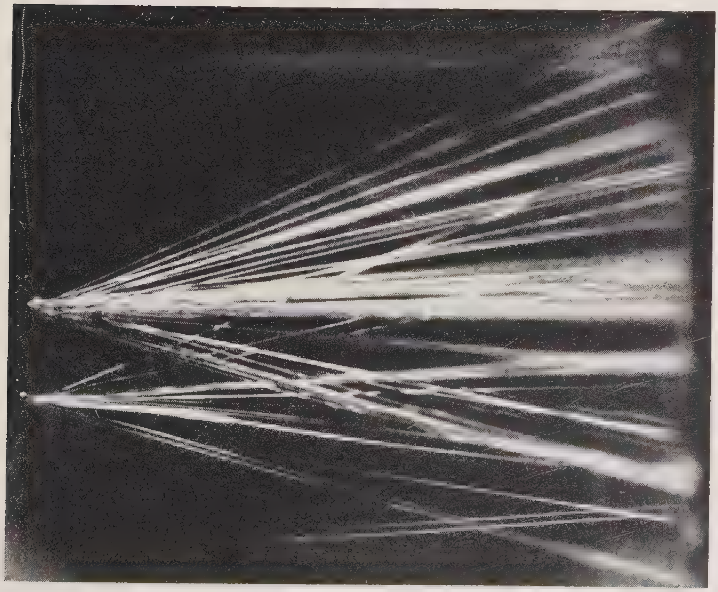

FIG. 2. Etch tubes terminating at inclusions (26×).

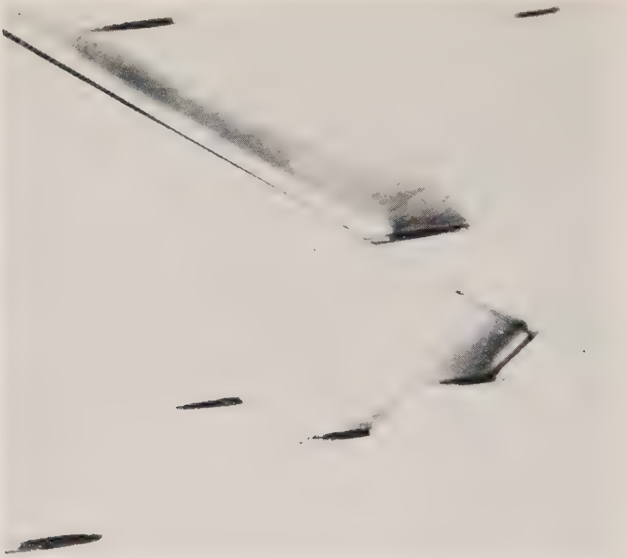

FIG. 3. Etched (1120) surface (100×).

the direction of the *c*-axis of the crystal. The nature of these pits maybe seen in Fig. 3 which shows seven of the pits from which tubes were formed. (The straight lines are cracks which will be discussed later.) The tubes extend below the pits in a ribbon-like form which is sometimes only about one micron wide. The ribbons curve in toward the seed plate in a random but smooth manner. Those ribbons pointing towards inclusions are straight.

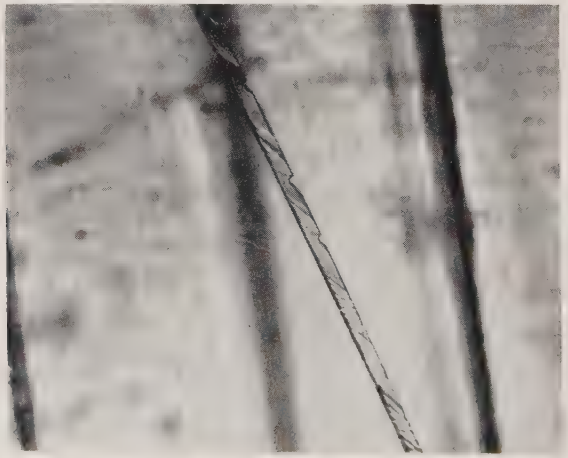

FIG. 4. Magnified etch tubes (67×).

The detailed structure of the ribbon-like tubes is somewhat difficult to photograph, but Fig. 4 shows a tube at 67× magnification. As may be seen the wall of the tube contains a group of parallel etch lines at an angle approximately 60° to the wall and a second group of lines at an approximate angle of 30°. Further observation disclosed that some tubes in addition to having parallel lines at different angles also have irregularly spaced sawtoothed profiles.

In Fig. 5A a typical ribbon-tube is sketched, directed from the surface toward the apex of a cone where an inclusion resides. From this sketch it may be noted that the wall of the tube appears to be discontinuous, an opaque part filling only about one third of the tube near the orifice and the outer edge of the tube being delineated by a single line boundary. In order to substantiate the capillarity of the tube a drop of carmine ink was applied to the surface and later a drop of xylene. Since the index of refraction of the xylene was sufficiently near that of the crystal, as penetration advanced the opacity of the tube lining completely disappeared, leaving the tube bounded by single lines. Upon allowing the xylene to evaporate the part opaque part transparent pattern in the tubes was re-established. Thus the transparent edges of the tubes were shown not to be trapped solvent but regions where the tubes were very thin and uniformly etched.

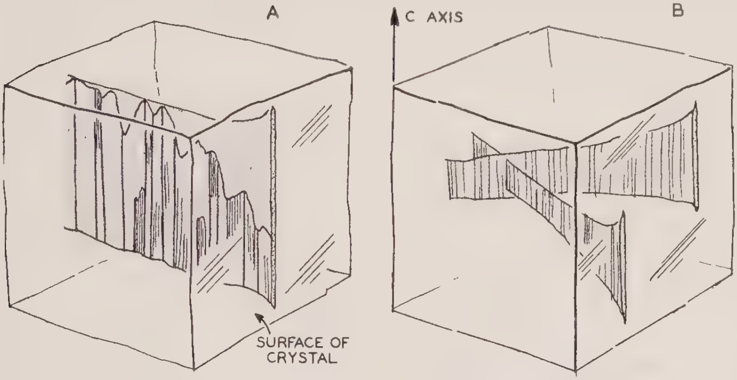

Fig. 5. Sketch of etch tubes originating at a (11$\bar{2}$0) face.

Occasional instances were noticed where two tubes had intersected. A sketch, Fig. 5B, illustrates one of these pairs. In both sketches it will be noted that the plane of the ribbon-like tubes was parallel to the *c*-axis of the crystal even though the angles the tubes make with the crystal surface are quite different.

It is interesting to compare the observations on this BTL Y-bar crystal with those which Arnold made on a Clevite Y-bar crystal. Arnold observed a much higher density of etch tubes in his crystal. Furthermore, the tubes he observed took a very irregular path from the $(11\bar{2}0)$ face to the seed whereas the BTL Y-bar crystal exhibited etch tubes whose gross forms were smooth curves. The crystals studied by the workers at Clevite appeared much like the one studied by Arnold. This difference probably arises because of the difference in growth conditions. Clevite Corporation uses a sodium carbonate solution to grow quartz while sodium hydroxide are used at these Laboratories. Furthermore, the temperatures and pressures used at these Laboratories exceed those used at Clevite Corporation. A Z-cut crystal grown at General Electric, Wembley, England, was also etched and found to have a much higher density of tubes than the BTL crystal, but the tubes traced smooth curves similar to those in crystal #2.

All synthetic Y-bar and Z-cut crystals, regardless of origin, exhibit a much higher density of etch pits on the slower growing $(11\bar{2}0)$ face, that is, the $-X$ direction. This is also the part of the crystal known to contain more impurities and which x-rays darken easily. Under the conditions of growth used at these Laboratories for crystal #2, one $(11\bar{2}0)$ face grew about four times as fast as the other. That is, the rate in $-X$ direction was $\frac{1}{4}$ that in the $+X$ direction. The density of etch tubes in the slow growing $(11\bar{2}0)$ face is about four times that in the opposite fast growing face.

Figure 6 shows a row of etch pits which appears on the slow growing $(11\bar{2}0)$ face of crystal #2. This row contains over 70 pits. None has an etch tube emanating from it. These pits recall those usually attributed to dislocations in metal crystals which are located at small angle boundaries. The long direction of the etch pits is parallel to the c-axis of the crystal as was observed in the case of the etch tubes. The presence of the striations and small peaks observed in this photograph is unexplained.

Finally, the cracks in crystal #2 were made by an accidental combination of thermal and mechanical shock. They all make angles close to 53° with the basal plane, corroborating the observations of Bloss[10] and Mrs. Wood[11], which indicate that quartz cleaves parallel to the rhombohedral faces. That this cleavage can be quite perfect is shown by the straight lines formed by the cracks in Fig. 3.

3. *CT Synthetic Crystal* (*BTL*)

As in the case of the Y-bar crystal, in the CT crystal (#3), the direction the tubes take from a given face is always determined by the growth direction. In this crystal the fast growing face was the minor rhombohedral, $(01\bar{1}1)$, face. The etch tubes were all directed toward the seed plate whose largest surface was the minor rhombohedral face. Some of the tubes, as in the case of the Y-bar crystal, terminate at inclusions of impurity. The tubes approach the seed plate at an angle which averages 79°, although angles as low as 67° and as high as 86° were measured. This places the majority of the tubes roughly parallel to one of the major rhombohedral faces.

A striking example of the propagation of the disturbance which leads to the etch tubes from seed plate to grown quartz was observed in crystal #3. Apparently the sodium hydroxide solution used in the hydrothermal method sometimes etches tubes in the quartz seed before growth begins. When growth does begin the etched tubes begin to fill in, but do so rapidly enough that inclusions of solvent are left in the seed. The disturbance causing etch tubes remains and is propagated as the crystal grows even though the surface of the seed appears to be sound. This is shown in Fig. 7 where a group of eight etch pits in the crystal forms the identical pattern of eight partially refilled tubes in the seed. (The seed crystal is the band bounded by two straight lines running from left to right in the lower half of the figure).

From Fig. 7 it can be seen that the tubes etched in the seed are not perpendicular to the seed plane; they make an angle with it about the same as that made by the etch tubes in the bulk of the crystal. The imperfections leading to the etch tubes in the seed and crystal do not lie in a straight line, however, as a close examination of Fig. 7 will show. The etch tubes formed by the HF, which are straight near their origin, make an angle with the seed plate 4° smaller than the angle made by the tubes in the seed with the seed plate. A microscopic examination of the tubes formed by the HF reveals that this 4° angle is caused by a gentle curving of the tubes near the seed and suggests that the etch tubes if elongated to the seed plate would meet the tubes in the seed plate at the same angle to the seed plane. The eight etch tubes were also observed on the other side of the seed.

In Fig. 8 the eight tubes appear with kinks near their origins. The kinks occur at that point because there the capping of the crystal which goes on during growth caused a major rhombohedral face (top of figure) to encroach on the domain of the minor rhombohedral growth. Although it is not obvious from the figure, the kinks in the tubes occur at the point from which they grew toward the $(10\bar{1}1)$ face instead of toward the $(01\bar{1}1)$ face, again demonstrating that the tubes have a strong tendency to follow the direction of growth.

It was also observed that many etch tubes terminated at inclusions (Fig. 7) as in crystal #2. Many terminated at the seed at points where no previously etched tubes could be found in the seed. Most of the tubes etched in the seed before growth had corresponding tubes etched in the crystal, but it could not be established that a tube existed in the crystal for every one in the seed.

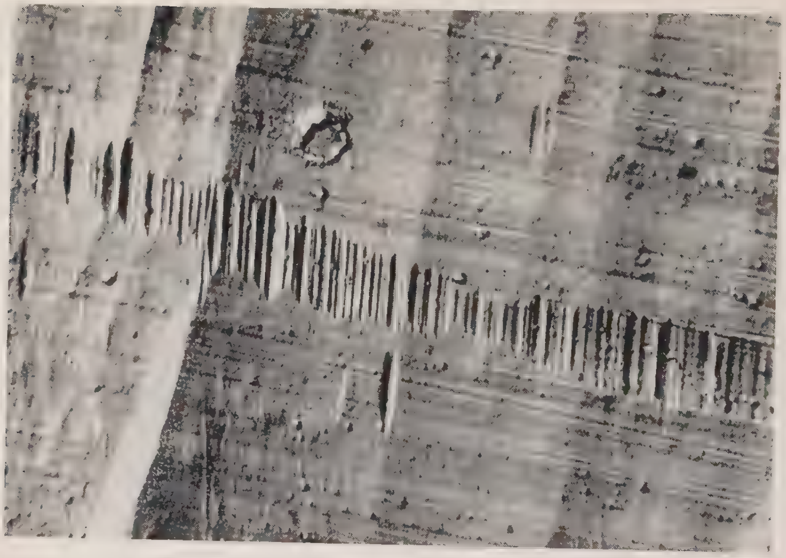

FIG. 6. Row of etch pits without tube formation (110×).

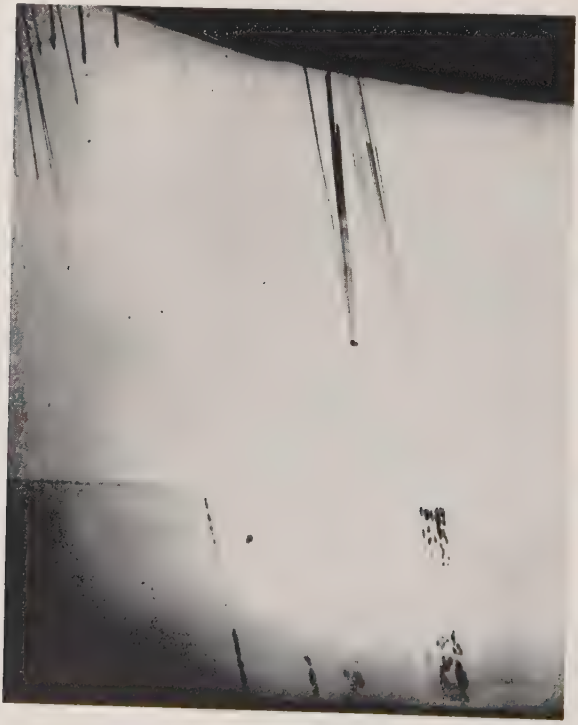

FIG. 7. Example of imperfections in seed continuing through grown crystal (13×).

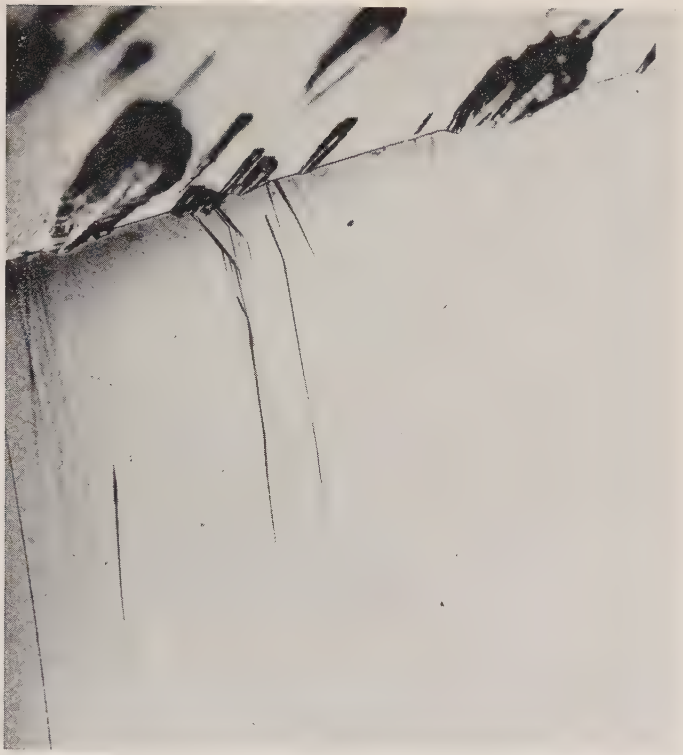

FIG. 8. Demonstration that etch tubes follow direction of growth (15×).

Finally, in no crystal of any type were etch tubes ever observed parallel, or nearly parallel, to the *c*-axis of the crystal.

DISCUSSION

It is apparent that natural and synthetic quartz contain imperfections which, in the presence of HF, cause etch pits to form. These pits are very deep compared to their width; indeed, the rate of solution along the length of the pit is sometimes 10^3 greater than that perpendicular to it. The nature of these imperfections is difficult to ascertain with the meager data presented here, but three possibilities are suggested.

First, the imperfections may be dislocations. If we disregard the difficulty of visualizing the form a dislocation takes in a structure such as quartz, and simply assume that a structure can be set up which satisfies the definition of a dislocation given by Read,[12] then one observation may suggest that the etch tubes are the result of dislocations. The one to one correspondence between partially refilled etch tubes in the seed plate and etch tubes in the grown crystal would be observed if the disturbance causing both were a dislocation in the seed. The dissolution of material around the dislocation in the seed would not "remove" the dislocation, for regrowth would generate it again and further growth would propagate it. This assumes, of course, that no other faults or disturbances occur as the crystal grows to refill the tube etched by the solvent. However, it is not impossible that the imperfections leading to the etch tubes in the seed were different from those causing them in the bulk of the crystal, and dislocations may not have been responsible for either. Thus one hesitates to state that the etch tubes were certainly the result of dislocations without more evidence. Unfortunately, quartz cannot be successfully deformed so that dislocations can be created and observed by decoration and etch in the normal manner. Furthermore, it seems risky to attribute etch tubes to dislocations if one considers the energy distribution necessary around such dislocations. In order to exhibit an etch rate along the length of the tube 10^3 times greater than that perpendicular to it, the lattice along the line of the dislocations would have to be violently disturbed. But the strain resulting from such a disturbance would have to be relieved a short distance from the line of disturbance or the rate of etch perpendicular to the line would also be rapid. The authors hesitate to suggest such a structure exists which would still be described as a dislocation.

A second explanation for formation of the etch tubes involves the nonuniform incorporation of impurities in the crystal as it grows without the formation of dislocations. If the crystal has a tendency to include impurities in pipes rather than uniformly, such pipes could serve as points of easy attack by the HF and the tubes would be formed by nothing more than the leaching out of the impurity. Some support for this point of view may be gleaned from the observation that the tubes are most numerous in faces known to contain the most impurities, such as the slow growing $(11\bar{2}0)$, and are absent in the material known to contain the least, the fast growing (0001) face. Cook[13] observed streaking and spots in a few monochromatic *x*-ray reflection photographs which he attributed to impurities, thus there is some evidence to show that these impurities are not uniformly distributed in the regions in which they occur. However, Cook found the streaks to be present in a $+X$ growth specimen and not in a $-X$ growth specimen, although $-X$ always contains more impurities. Cook did find that in general the line breadth was greater in the $-X$ growth specimens, indicating more defects of some type. Furthermore, smoky regions which are caused by impurities do not always exhibit etch tubes. Thus, if an impurity is responsible for the etch tubes it is apparently not the impurity which is considered to be responsible for smokiness.[1] Hydrogen, present as hydroxide, is one impurity which could cause etch tubes, but one is hard put to explain why hydroxide ion should not be incorporated into the lattice uniformly. One must, it seems, consider the nonuniform inclusion of impurities as only a possible cause of the formation of etch tubes.

A third explanation of the formation of the etch tubes is that they are the result of a combination of the two possibilities discussed above; i.e., they are caused by the etching of impurities which have precipitated along dislocation lines after diffusing to them. This would account for the great depth and small cross section of the etch tubes. Unfortunately, an attempt to isolate impurities from the etchant failed. Either the concentration of impurities was too small to be observed or the impurity formed a volatile fluoride, such as HF, which was lost.

If we do not attempt an atomistic picture of the defects resulting in etch tubes some aspects of the tubes themselves are interesting. For instance, why should the eight tubes shown in Fig. 7 make a slight curve near the seed which changes their direction of approach to the seed by 4°? One explanation is that because of a higher impurity content near the seed the ratio c/a for the quartz unit cell is slightly different there. This should be detectable by careful x-ray analysis, but attempts by W. L. Bond[14] to observe lattice parameter differences between seed plates and grown quartz on other crystals have so far been unsuccessful.

The directions the etch tubes take are also puzzling. Although they tend to lie in the general direction of growth, they appear to be located at random on the face of origin and to make a multitude of angles with it. In the case of the Y-bar crystal all tubes pointing toward the seed were curved; all tubes pointing toward inclusions were straight. This was true even though the seed and inclusions were in the same region of the crystal. It is very difficult to suggest a mechanism for tube formation which explains this.

Equally puzzling are the great differences observed in the density of etch tubes on different faces and on faces which differ only because quartz is piezoelectric. Thus no etch tubes are observed lying near the (0001) direction in any crystal, and $(11\bar{2}0)$, $(-X)$, always has a high density of etch tubes in Y-bar crystals but none in CT crystals. From the few examples studied one gets the impression that the density of etch tubes is related to the impurity content which, in turn, depends in some way on the polarity of the crystal and perhaps direction of growth. It is known that the purity of quartz grown on various faces, beginning with Y-bar seeds, varies as follows in order of decreasing impurity content: $[11\bar{2}0]\ (-X) > [11\bar{2}0](+X) > [0001]$. The density of etch tubes observed on the faces varies in the same manner. BTL synthetic quartz exhibited pit densities of the order 10 cm^{-2} while the natural crystal had a higher density of the order 10^4 cm^{-2}. These estimates are crude and do not apply to the same crystallographic face. The intersection of two etch tubes which was observed at several points in crystal #2 is another phenomenon which is not explained. If the tubes are caused by dislocations the lines in this case must intersect without disturbing each other. It is hard to believe that two dislocation lines lying in two different but nonspecific directions would intersect without some change in one or the other, or both, being observed.

With so many questions unanswered and so few samples, the authors offer no single explanation for the origin and behavior of the etch tubes. It is hoped that the work done so far will stimulate further research. For instance, it is apparent that the preparation of an ultra-pure quartz crystal is of paramount importance. Then etching experiments and experiments like those conducted by Cook,[13] and King,[2] could be done with the hope of discerning the role played by trace impurities in modifying the properties of quartz crystals.

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INFRARED STUDY OF THE CARBONATE MINERALS

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Abstract

Infrared spectra of 27 common and rare carbonates are described. Each carbonate shows characteristic absorption bands, some of which differ more or less from published curves. Among the isomorphous members of the calcite, dolomite, and aragonite groups, a noteworthy shift of absorption bands takes place with longer wave lengths corresponding to an increase in cation radius or mass. The spectral difference between the more common groups may be related to crystal structure. The spectral relationships among the minerals of several groups are not well known, due to the complicated composition and crystal structure. Infrared active groups of CO_3, HCO_3, H_2O, OH, and SO_4 dominate the absorption characteristics.

Introduction

The infrared spectra of anhydrous carbonates among minerals and chemical compounds have been studied to some degree by Schaefer and Schubert (1916), Schaefer et al. (1926), Menzies (1931), Halford (1946), Hunt et al. (1950), Adler et al. (1950), Miller and Wilkins (1952), Keller et al. (1952), and Hunt and Turner (1953). Yet a comprehensive infrared survey of the various carbonate mineral groups using a precision spectrophotometer does not appear to have been made.

In this study it is proposed to furnish systematic data on the infrared spectra of these carbonate minerals, and to infer in general terms the correlation between spectral changes and internal structure. The interpretation is incomplete because of the limited number of samples of satisfactory purity and the partial knowledge of the crystal structure of several carbonates. However, a distinct spectral relationship may be demonstrated among members of isomorphous groups, while both major and minor absorption bands appear to be diagnostic for each mineral.

Procedure

The samples examined were obtained primarily from the mineral collections of Columbia University, supplemented by specimens from the American Museum of Natural History, New York, and the United States National Museum, Washington, D. C. (Table 1). All were checked optically and trona, kutnahorite, gaylussite, schroeckingerite, and andersonite were examined by *x*-ray diffraction (Table 1). Samples containing recognizable impurities were eliminated. Infrared specta were obtained with a Perkin-Elmer Model 21 double beam recording infrared

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* Visiting Professor from National Taiwan University, 1958–59.

TABLE 1. CARBONATE MINERALS EXAMINED

No.	Minerals and Groups*	Optics Measured
	Acid Carbonates	
5	Nahcolite, $NaHCO_3$—Mon. Rifle, Colo. (Am.M. 23281**)	$\alpha < 1.410$, γ: 1.582, (−)2V large
6	Trona, $Na_3H(CO_3)_2 \cdot 2H_2O$—Mon. Owens Lake, Calif.	α: 1.415, γ: 1.540, $\gamma - \alpha = 0.125$, (−)2V large
	Anhydrous Normal Carbonates	
	Calcite Group	
7	Magnesite, $MgCO_3$—Hex.R. Chewelah, Wash.	ω: 1.690, ϵ: 1.602, Uniax.(−)
8	Smithsonite, $ZnCO_3$—Hex.R. Magdalena, N. M.	ϵ: 1.730, Uniax.(−)
9	Siderite, $FeCO_3$—Hex.R. Westphalia	ϵ: 1.713, Uniax.(−)
10	Rhodochrosite, $MnCO_3$—Hex.R. Butte, Mont.	ϵ: 1.692, Uniax.(−)
11	Calcite, $CaCO_3$—Hex.R. Butte, Mont.	ω: 1.660, ϵ: 1.570, Uniax.(−)
12	Calcite, $CaCO_3$—Hex.R. Mineral Point, Wis.	ω: 1.662, ϵ: 1.568, Uniax.(−)
	Dolomite Group	
13	Dolomite, $CaMg(CO_3)_2$—Hex.R. Senator Mine, Santa Clara Co., Calif.	ω: 1.680, ϵ: 1.590, Uniax.(−)
14	Ankerite, $Ca(Fe, Mg)(CO_3)_2$—Hex.R. Brazil	ω: 1.695, ϵ: 1.602, Uniax.(−)
15	Kutnahorite, $Ca(Mn, Mg)(CO_3)_2$—Hex.R. Franklin, N. J. (Am.M. 30630)	ω: 1.742, ϵ: 1.630, Uniax.(−)
	Aragonite Group	
16	Aragonite, $CaCO_3$—Ort. Alameda Co., Calif.	α: 1.527, γ: 1.680, $\gamma - \alpha = 0.153$, (−)2V small
17	Aragonite, $CaCO_3$—Ort. Ural Mts., U.S.S.R.	α: 1.525, γ: 1.675, $\gamma - \alpha = 0.150$, (−)2V small
18	Aragonite, $CaCO_3$—Ort. Livermore, Calif.	α: 1.525, γ:1.680, $\gamma - \alpha = 0.155$, (−)2V small
19	Strontianite, $SrCO_3$—Ort. Dreusteinfurt, Westphalia	α: 1.520, γ: 1.666, $\gamma - \alpha = 0.146$, (−)2V small
20	Witherite, $BaCO_3$—Ort. Alston Moor, England	α: 1.530, γ: 1.677, $\gamma - \alpha = 0.147$, (−)2V small
21	Cerussite, $PbCO_3$—Ort. Ibbenburen, Westphalia	$1.82 > \alpha > 1.780$, (−)2V small
	Hydrated Normal Carbonates	
22	Thermonatrite, $Na_2CO_3 \cdot H_2O$—Ort. Vesuvius, Italy	α: 1.425, γ: 1.523, $\gamma - \alpha = 0.098$
23	Pirssonite, $Na_2Ca(CO_3)_2 \cdot 2H_2O$—Ort. Searles Lake, Calif. (Am.M. 26198)	α: 1.504, γ: 1.572, $\gamma - \alpha = 0.068$, (+)2V small
24	Gaylussite, $Na_2Ca(CO_3)_2 \cdot 5H_2O$—Mon. Salt Pen, Pretoria	α: 1.450, γ: 1.520, $\gamma - \alpha = 0.070$, Biax.(−)
25	Gaylussite, $Na_2Ca(CO_3)_2 \cdot 5H_2O$—Mon. Ragtown, Nev.	α: 1.450, γ: 1.520, $\gamma - \alpha = 0.070$, Biax.(−)
26	Schroeckingerite, $NaCa_3(UO_2)(CO_3)_3(SO_4)F \cdot 10H_2O$—Ort. Lost Creek, Wamsutter, Wyo.	α: 1.493, γ: 1.540, $\gamma - \alpha = 0.047$, (−)2V small
27	Andersonite, $Na_2Ca(UO_2)(CO_3)_3 \cdot 6H_2O$—Hex.R. Atomic King No. 2, near Moab, Utah (Nat.M. 107414†)	γ: 1.522, ϵ: 1.540, $\epsilon - \omega = 0.018$, Uniax.(+)
	Carbonates with Hydroxyl or Halogen	
28	Zaratite, $Ni_3(CO_3)(OH)_4 \cdot 4H_2O$—Iso.? Wood's Mine, Penn.	n: 1.588–1.600, some show double refraction
29	Malachite, $Cu_2(CO_3)(OH)_2$—Mon. Globe, Ariz.	α: 1.650
30	Azurite, $Cu_3(CO_3)_2(OH)_2$—Mon. Copper Queen, Ariz.	α: 1.728, $\gamma > 1.83$
31	Northupite, $Na_3Mg(CO_3)_2Cl$—Iso. Searles Lake, Calif. (Am.M. 22041)	n: 1.513

* Unless otherwise indicated specimens are from the Columbia University collection. Groups are after Palache, C., Berman, H., and Frondel, C. (1951), The system of mineralogy, 2, 7th ed., John Wiley & Sons, Inc., New York, 132.

** Am.M.: American Museum of Natural History, New York.

† Nat.M.: United States National Museum, Washington, D. C.

ϵ: Index on cleavage.

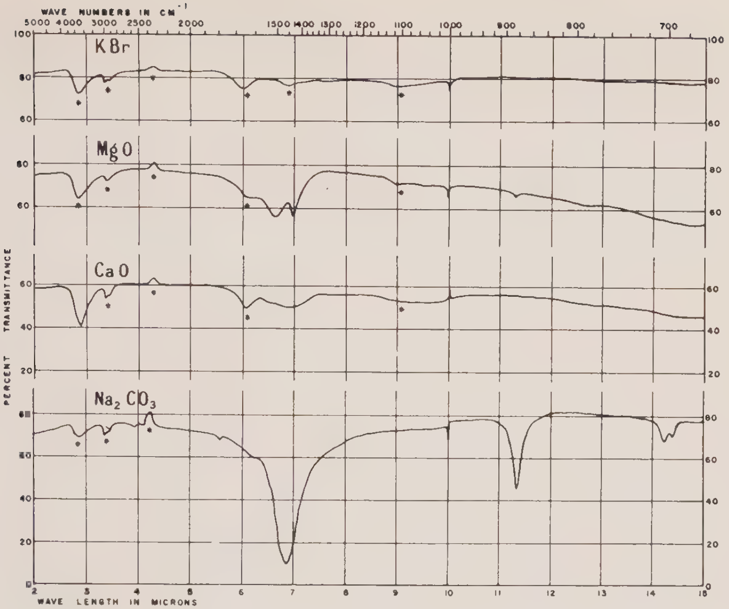

FIG. 1. Infrared spectra of reference chemical compounds.

spectrophotometer, using a NaCl prism through the spectral range 2 to 15 microns.

The method of sample preparation for infrared examination corresponds to the procedure by French et al. (1954) using the pressed pellet technique. In order to reduce light scattering, which causes a decrease in transmission and the degree of resolution, each sample was ground to less than 300 mesh in an agate mortar. A 400 to 500 mg. KBr plate, 13 mm. in diameter and 1 mm. thick, containing 1–2 mg. of the sample and compressed under a force of 2,200 lbs. on a disc 13 mm. in diameter, was found to be most satisfactory.

INFRARED SPECTRA

Reference chemical compounds

The spectra of the significant reference compounds KBr, MgO, CaO, and Na_2CO_3 are compared in Fig. 1, while the approximate numerical positions of absorption bands and corresponding intensities of absorption are listed in Table 2. Potassium bromide shows six absorption bands of medium to weak intensity, which include peaks at 2.85 and 6.00 microns caused by absorbed moisture, also two small shoulders at 4.15 and 4.35 microns. In the infrared curves shown, where KBr is used as a

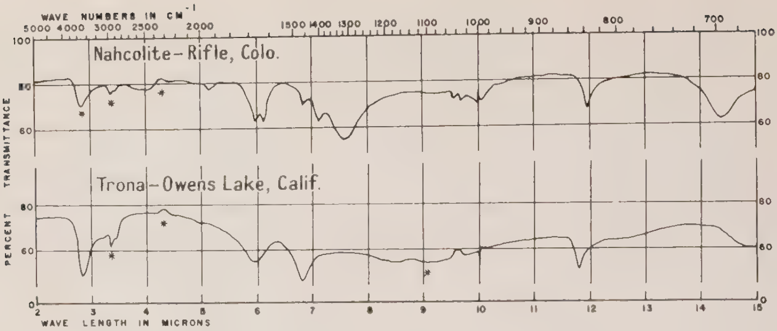

Fig. 2. Infrared spectra of acid carbonates.

mounting medium, the corresponding effects are eliminated in the tables and the peaks influenced in the graphs are marked with asterisks.

Magnesium oxide has sharp absorption peaks at 6.70 and 7.00 microns and weak bands at 11.30 and 12.50 microns (Fig. 1). Calcium oxide shows a single weak broad band at 6.70–7.00 microns, with a strong peak at 2.85 microns caused by absorbed water. The spectra of magnesite and calcite (Fig. 3) show that bands characteristic of the oxides are weak or absent, indicating a greater influence on the curves of infrared active CO_3 groups. Sodium carbonate shows strong absorption at 6.90 and 11.35 microns and weak bands at 3.95, 5.57, 14.25, and 14.40 microns caused by CO_3, which suggest the spectra of the aragonite group (Fig. 4).

Table 2. Absorption Bands of the Reference Chemical Compounds

No.	Chem. Compound	Positions of Absorption Bands*									
1	KBr	2.85M 3510	3.35W 2990 3.45W 2900	4.15W 2410 4.35W 2300		6.00W 1667 6.87W 1456		9.02W 1110			
2	MgO					6.70M 1493	7.00M 1429		11.30W 885	12.50W 800	
3	CaO	2.85S 3510				6.70– 1493–	7.00W 1429				
4	Na_2CO_3		3.95W 2530		5.57W 1795	6.90S 1450			11.35S 881		14.25W 702 14.40W 694

* Wave numbers (cm⁻¹) are given below the corresponding wave length values (μ). S: strong, M: medium, W: weak.

Acid carbonates

In this group the spectra of nahcolite and trona show a partial similarity in absorption, although the structural relationships between the two are not well known (Fig. 2 and Table 3). In trona, two strong peaks caused by water of crystallization appear at 2.83 and 5.92 microns. In nahcolite, which has no molecular water, double maxima occur around 6 microns, which suggest some hydration during sample preparation (Keller et al., 1952). Absorption bands caused by the HCO_3 groups seem to occur near 5 and 7 microns, in addition to those near 10, 12, and 14 microns (Miller et al., 1952).

Table 3. Absorption Bands of the Acid Carbonates

No.	Mineral	Positions of Absorption Bands						
5	Nahcolite		5.15W	6.13M	7.10M	9.55W	11.95M	14.40S
			1940	1630	1409	1047	837	694
			5.97M	6.85W	7.60S	9.66W		
			1675	1460	1316	1035		
6	Trona	2.83S	5.92M	6.80S		9.45W	11.75M	14.70M$^-$
						1058		
						9.65W		
		3530	1690	1472		1037	851	681 −

Calcite and dolomite groups

The infrared spectra of the calcite and dolomite groups are characterized by three prominent absorption maxima at 6.90–6.97, 11.28–11.55, and 13.36–14.02 microns and two minor peaks at 3.92–3.97 and 5.47–5.52 microns, as shown in Fig. 3 and Table 4. These bands are all considered to be caused by the CO_3 groups in the crystals. Although the minor absorption bands are poorly expressed in some spectra, they appear when satisfactory resolution is achieved. The consistent character of the infrared spectrum for a mineral species is illustrated by the two samples of calcite from different localities, which show essentially identical absorption bands. Halford (1946) has determined from an analysis of space group characteristics the existence of four molecular frequencies for the CO_3 ions in calcite, of which three are active in the infrared spectrum, also of six infrared active molecular frequencies in aragonite.

The similarity of the minerals of the dolomite group to the calcite group in atomic structure (Bragg, 1914; 1937; Wyckoff and Merwin, 1924) is reflected in the infrared spectra indicated above, as well as two absorption bands of dolomite between 11 and 12, and 13 and 14 microns

TABLE 4. ABSORPTION BANDS OF THE CALCITE AND DOLOMITE GROUPS

No.	Mineral	Positions of Absorption Bands						
7	Magnesite		5.50W	6.90S		11.28S	13.36M	
			1818	1450		887	748	
8	Smithsonite	3.95W	5.47W	6.95S		11.50S	13.45M	
		2530	1830	1440		870	743	
9	Siderite		5.50W		7.03S	11.55S	13.58M	
			1818		1422	866	737	
10	Rhodochrosite	3.95W	5.52W	6.98S		11.53S	13.76M	
		2530	1810	1433		867	727	
11	Calcite	3.93W	5.52W	6.97S		11.42S		14.03M
		2545	1812	1435		876		712
12	Calcite	3.92W	5.50W	6.97S		11.40S		14.02M
		2550	1818	1435		878		713
13	Dolomite	3.95W	5.50W	6.90S		11.35S	13.70M	
		2530	1818	1450		881	730	
14	Ankerite	3.95W	5.48W	6.90S		11.40S	13.77M	
		2530	1825	1450		877	726	
15	Kutnahorite	3.97W	5.50W	6.97S		11.50S	13.87M	
		2520	1818	1435		869	721	

which lie between those of calcite and magnesite. These two absorption bands appear to be shifted to longer wave lengths as the divalent cations in the mineral increase in atomic radius, in the order magnesite, smithsonite, siderite, rhodochrosite, and calcite in the calcite group, and dolomite, ankerite, and kutnahorite in the dolomite group (Fig. 3). This relationship has been mentioned by Adler et al. (1950) and Keller et al. (1952). The bands between 13 and 14 microns are the most diagnostic and have been applied in both qualitative and quantitative determination of the mineral constituents of rocks (Hunt and Turner, 1953). It is conceivable that the longer inter-atomic distances caused by larger cations may result in lower frequencies, that is, longer wave lengths.

Aragonite group

In the minerals of the aragonite group, major absorption peaks appear at 6.70–7.13, 11.40–11.90, and 14.03–14.77 microns, and minor bands at

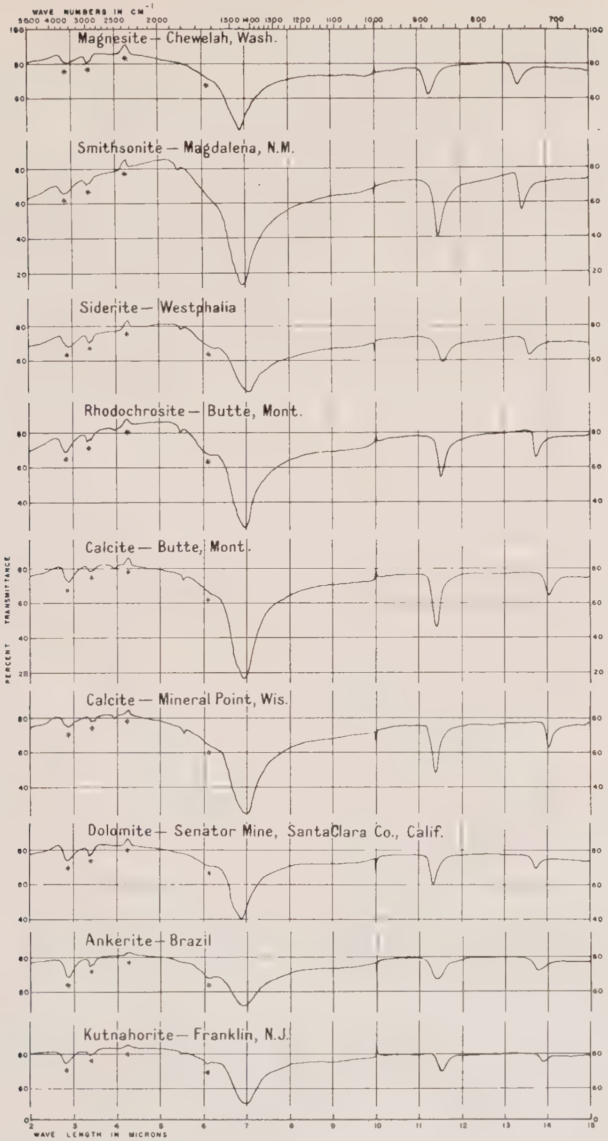

FIG. 3. Infrared spectra of the calcite and dolomite groups.

3.93–4.00, 5.53–5.75, and 9.22–9.48 microns (Fig. 4 and Table 5). The absorption bands between 11 and 12 and between 14 and 15 microns show double maxima in aragonite, but only one peak in witherite and cerussite. Strontianite has one peak at 11–12 microns and double bands at 14–15

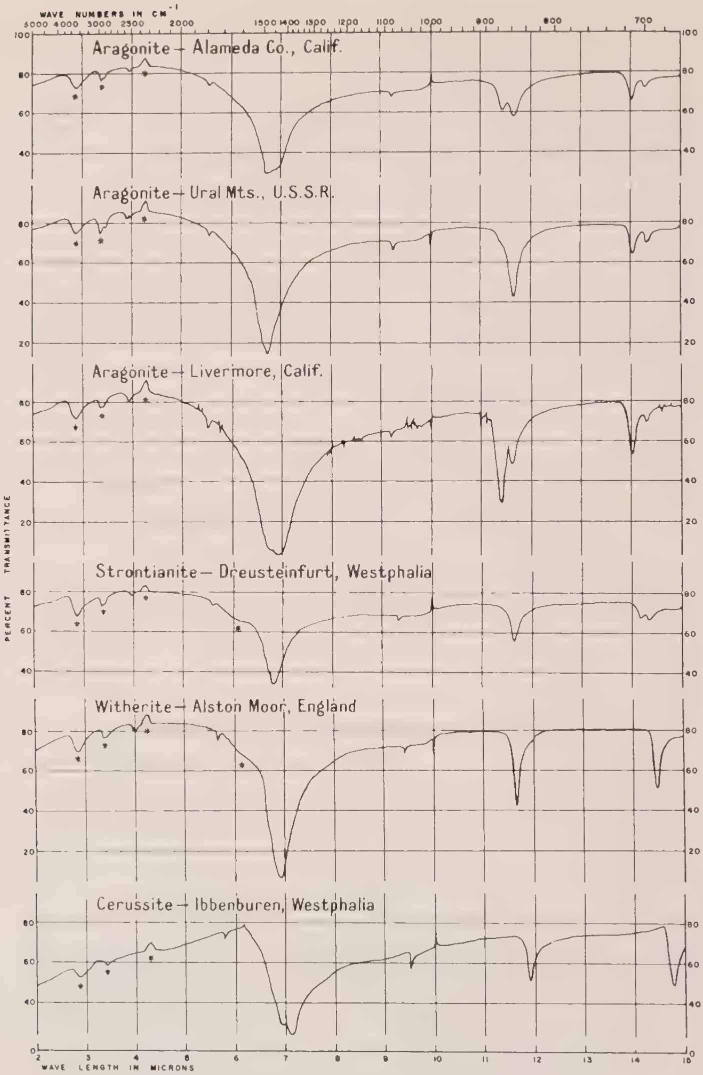

FIG. 4. Infrared spectra of the aragonite group.

microns, somewhat intermediate between aragonite and witherite. The strongest band near 7 microns appears essentially as a broad band in aragonite, and splits into two peaks in cerussite. Three spectra of aragonite from different localities are consistent in major features, although they differ moderately in the intensity of absorption in some maxima. Minute protuberances in the curve of aragonite from Livermore, Califor-

TABLE 5. ABSORPTION BANDS OF THE ARAGONITE GROUP

No.	Mineral	Positions of Absorption Bands						
16	Aragonite	3.95W		5.55W	6.70 –7.00S	9.22W	11.42M	14.03M
		2530		1803	1493 –1429	1085	875	712
							11.63S	14.30W
							860	699
17	Aragonite	3.95W		5.55W	6.72S	9.22W	11.40W	14.03M
		2530		1803	1490	1085	87	712
							11.63S	14.30W
							860	699
18	Aragonite	3.93W		5.53W	6.70 –7.00S	9.22W	11.42S	14.03M
		2545		1808	1493 –1429	1085	875	712
							11.63M	14.30
							860	699
19	Strontianite	3.97W		5.57W	6.80S	9.30W	11.63S	14.15W
		2520		1795	1470	1075	860	707
								14.30W
								699
20	Witherite		4.00W	5.65W	6.92S	9.40W	11.63S	14.43S
			2500	1770	1445	1064	860	693
21	Cerussite			5.75W	6.95S 7.13S	9.48W	11.90M	14.77S
				1740	1440 1404	1055	841	677

nia are probably caused by impurities. The infrared spectra obtained on common carbonates by previous workers (Adler et al., 1950; Keller et al., 1952) show somewhat different positions for the absorption bands, but the minor peaks are incompletely shown, which may be attributed to the purity of the samples or the sensitivity of the instrument.

The shift of the absorption bands is more apparent than in the calcite and dolomite groups. Apparently it follows the sequence of increase in atomic weight rather than atomic radius. The strong bands in the areas 11–12 and 14–15 microns and also other maxima, including even minor peaks, are shifted to longer wave lengths, as the cations in the mineral become heavier, in the order of aragonite, strontianite, witherite, and cerussite. Also, the larger cations of the minerals of this group cause the absorption bands to occur at longer wave lengths between 14 and 15, rather than between 13 and 14 microns, as in the calcite-dolomite group. The condition that larger atomic masses may yield to longer wave lengths arises from the equation

$$v=\frac{i}{2\pi c}\sqrt{\frac{k}{u}},$$

where v = frequency of vibration in cm^{-1}, c = velocity of light, u = reduced mass of the vibrating atoms, k = force constant (Barnes et al., 1944).

A comparison of the spectra indicates that the positions of the absorption bands are affected by the cations present, but the spectra are dominated by the CO_3 groups. In the spectra of a calcite and dolomite groups, the weak absorption bands between 9 and 10 microns are absent, and no double peaks appear. This difference may be explained by the structural difference, since in rhombohedral calcite the CO_3 group is placed so that each oxygen atom is in contact with two calcium atoms, while in orthorhombic aragonite each oxygen atom is in contact with three calcium atoms (Bragg, 1937; Adler et al., 1950; Keller et al., 1952).

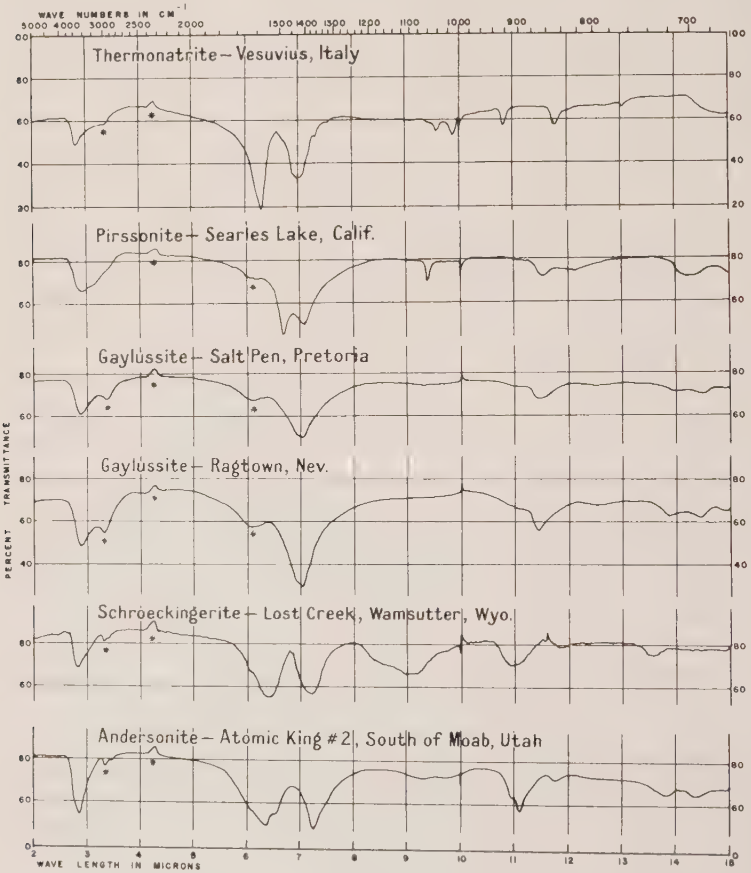

FIG. 5. Infrared spectra of hydrated normal carbonates.

Observations on carbonate mixtures were made by Keller et al. (1952) on artificial mixtures of calcite with aragonite, and calcite with magnesite. The curves of the mixtures were noted to contain all the peaks characteristic of the spectrum of each individual constituent. However, differences were observed between the spectra of dolomite and a calcite-magnesite mixture, since the former is a distinct mineral species, while the latter is a mechanical mixture.

Hydrated normal carbonates

The infrared spectra of the hydrated normal carbonates (Fig. 5 and Table 6) are characterized by a range in position and intensity of bands caused by H_2O molecules. The absorption peaks caused by water of crystallization are shown at 2.82–2.90 and 6.28–6.52 microns, and the maxima attributed to the CO_3 groups at 6.70–7.22 and between 11 and 15 microns. In addition, a characteristic absorption band caused by the SO_4 radical appears at 9.12 microns in schroeckingerite. A silicate impurity appears to have been present in the samples of thermonatrite and pirssonite, resulting in small peaks between 9 and 11 microns (Miller et al., 1952; Keller et al., 1952). The two spectra of gaylussite from different localities show closely similar absorption bands.

TABLE 6. ABSORPTION BANDS OF THE HYDRATED NORMAL CARBONATES

No.	Mineral	Positions of Absorption Bands								
22	Thermonatrite	2.82M 3540	6.28S 1592	7.00S 1429 7.28W 1373		9.57W 1045 9.85W 1016	10.82W 924	11.75W 851	13.02W 768	
23	Pirssonite	2.85M 3510	6.70S 1493	7.05S 1419		9.35W 1070		11.53W 867		14.25W 702
24	Gaylussite	2.85M 3510		7.05S 1419				11.45W 873	13.92W 718	14.48W 691
25	Gaylussite	2.90S 3450		7.05S 1419				11.42M 876	13.92W 718	14.45W 692
26	Schroeckingerite	2.85M 3510	6.42S 1558	7.20S 1390	8.62W 1160	9.12M 1098	10.95M 913	11.85W 844	13.55W 738	
27	Andersonite	2.85S 3510	6.33S 1580 6.52M 1535	7.22S 1385		9.27W 1080	10.95M 913	11.10S 901 11.80W 848	13.75W 727	14.35W 697

The carbonate minerals of this group are more complicated in chemical composition and crystal structure, and the relationships between the infrared spectra and atomic structure are not well known. However, the similarity of schroeckingerite and andersonite in infrared spectra suggests a possible structural analogy between the two uranium minerals. Schroeckingerite is orthorhombic, pseudo-hexagonal in form (Hurlbut,

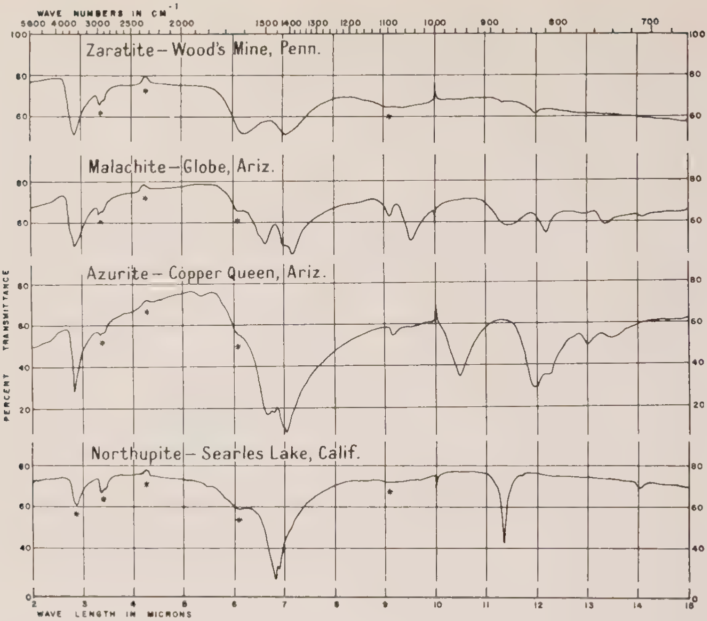

FIG. 6. Infrared spectra of carbonates containing hydroxyl or halogen.

1954), and andersonite shows a rhombohedral lattice (Axelrod et al., 1951). Infrared curves are somewhat alike, but the *X*-ray diffraction patterns differ.

Carbonates containing hydroxyl or halogen

The absorption bands of four representatives of this group, zaratite, malachite, azurite, and northupite, are shown in Fig. 6 and Table 7. Characteristic strong maxima due to hydrogen bonds (Keller and Pickett, 1950) appear at 2.85 microns. In zaratite a band caused by water of crystallization occurs at 6.25 microns, in addition to a band caused by hydroxyl bonding. The absorption bands caused by the CO_3 groups occur chiefly near 7 microns as strong peaks and at wave lengths longer than 9 microns as two or more maxima of varying intensity. In azurite and northupite a weak band occurs at 5.40–5.47 microns, comparable to bands yielded by the minerals of the calcite and dolomite groups, while in malachite and azurite a weak peak occurs at 9.10–9.15 microns similar to the bands given by the minerals of the aragonite group (see Figs. 3 and 4).

The spectral relation to crystal structure is not well known among the minerals of this group. Malachite belongs to the space group $P2_1/a$ (Ramsdell and Wolfe, 1950), and azurite to $P2_1/c$ (Brasseur, 1932). The

Table 7. Absorption Bands of the Carbonates Containing Hydroxyl or Halogen

No.	Mineral	Positions of Absorption Bands									
28	Zaratite	2.85S 3510		6.25S 1600	7.05S 1419			11.35W 881 11.97W 835			
29	Malachite	2.85S 3510		6.62S 1510	7.00M 1430 7.17S 1395	9.10W 1099 9.52S 1050		11.45M 873	12.17M 822 12.90W 775	13.33W 750	14.05W 712
30	Azurite	2.84S 3520	5.40W 1852	6.63S 1509 6.78W 1475	7.03S 1422	9.15W 1093	10.47S 955	11.97S 835	12.23M 817	13.00W 769 13.45W 743	
31	Northupite		5.47W 1830	6.78S 1475 6.85S 1461				11.33S 882			14.02W 713

general similarity in crystal structure which at the same time includes minor differences is reflected in the infrared spectra. The absorption bands of northupite correspond to those of the calcite and dolomite groups, except for the doublet near 7 microns. It is worthy of note that the bands at 11.33 and 14.02 microns are located between corresponding bands for sodium carbonate and magnesite (see Figs. 1 and 3). The influence of chlorine on the spectrum appears to be subordinate, as in the case of the cations.

Conclusion

The infrared spectra of the calcite and dolomite groups are characterized by three major and two minor absorption bands attributed to CO_3, while those of the aragonite group exhibit three to five major and three minor peaks, according to the difference in crystal structure. There is also a definite relation between band shift and the atomic radius or mass of the cations contained in these isomorphous groups.

The minerals of other groups are more complicated in chemical composition and crystal structure, and the mutual spectral relationships are as yet indefinite. However, the absorption bands caused by the infrared active groups, such as CO_3, HCO_3, H_2O, OH, and SO_4, are revealed, and the spectrum is diagnostic for each mineral.

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The writers wish to express their appreciation to Dr. Brian H. Mason of the American Museum of Natural History, New York, and to Dr. A. C. Smith of the United States National Museum, Washington, D. C., for furnishing certain carbonate specimens for the present study. The cooperation of Messrs. W. A. Bassett and G. H. Megrue in discussion of the manuscript is greatly appreciated.

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ERROR ANALYSIS FOR THE BUERGER PRECESSION CAMERA*

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ABSTRACT

A routine for the reduction of the data from precession *x*-ray diffraction photographs leads to a direct estimate of the reliability of the cell constants calculated from such measurements. Calibration routines are also discussed.

1. INTRODUCTION

The Buerger precession camera (Buerger, 1944) is a very useful instrument for the rapid determination of the cell dimensions and the space group of a single crystal. Although back reflection and other techniques may exceed the precession camera in precision by one or two powers of ten, the latter permits measurements which are quite accurate enough for the cataloging of single crystal cell data (cf. Donnay and Nowacki, 1954) and for the recognition of data previously cataloged. It is also accurate enough to provide cell dimensions which are adequate for the conversion of the results of most modern structure analysis into molecular dimensions.

The accuracy which can be attained by the use of this instrument has been discussed by several authors (Evans *et al.*, 1949; Barnes, 1949; Evans, 1949; Barnes *et al.*, 1951). This paper is written to present a routine for the reduction of the data from precession photographs which indicates the precision attained in the measurement of a given photograph or set of photographs. We find that under ideal conditions, the precision attainable is higher than that suggested by previous writers. Under non-ideal conditions the method of reduction delivers a numerical estimate of the degree of precision which has been achieved. The need for the rediscussion of the accuracy of the camera became apparent in the course of a study of the cell dimensions of a group of alkali citrates (Love and Patterson, 1960).

In a precession photograph taken of a zero layer reciprocal lattice net set accurately parallel to the flat film, the *x*-ray diffraction spots occur in parallel rows and the net formed by these spots is an image of the reciprocal lattice net in question. The separation X (Fd^* according to Buerger,

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1944) of two parallel rows on the film is related to the reciprocal A of the distance between the corresponding rows of the reciprocal lattice by the relation*

$$A = \frac{\lambda F S_m}{X S} \tag{1}$$

in which λ is the wavelength of the radiation, F is the "magnification factor," *i.e.*, the crystal to film distance, X is the measured distance between the rows, S is the distance between the two holes in the cassette which are used to produce fiducial spots on the film. These are found to have a separation S_m when measured at the time the quantity X is determined (see section 3), and permit the correction for shrinkage implied by the ratio S_m/S in (1).

Three of the quantities in (1) *i.e.*, λ, F, S can be eliminated from consideration provided that the camera is calibrated by means of a crystal of known A_0. In this calibration we observe rows of separation X_0 on a film for which the measured distance between the fiducial spots is S_{m0}. In such a case, the quantity A is obtained from

$$A = \frac{X_0}{X} \frac{S_m}{S_{m0}} A_0 \tag{2}$$

provided that the same wavelength is used both for calibration and for the measurement. If two different wavelengths are used, λ_0 and λ respectively, the expression

$$A = \frac{\lambda}{\lambda_0} \frac{X_0}{X} \frac{S_m}{S_{m0}} A_0 \tag{3}$$

must be used.

As is well known from statistical theory, the squared fractional standard deviation in A, i.e., $(\sigma A/A)^2$ as calculated from any of the formulae (1)–(3) is simply the sum of the squared fractional standard deviations of the quantities which enter into the particular formula. An analysis of the expected contribution from each source of error is given in Table 1 and the basis for the estimate of the contribution of each is discussed in the remarks in the table. The only measurements referred to in the table which require further comment are the linear measurements on the film and the way in which they are carried out. These depend on the least count M_0 of the measuring instrument used for this purpose. A discussion of the method we have used for the reduction of our data is given on page 328 and for the measurement of the shrinkage correction on page 329. We comment on the method used for the combination of observations

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* The quantity A is either a translation of the crystal lattice or is simply related to such a translation. See section 6.

For definitions see text.

Nature of Quantity	Symbol	Remarks	Squared Fractional Standard Deviation × 10^8					
				M = 0.05 mm.			M = 0.025 mm.	
			Equation #	(1)	(2)	(3)	(4)	(5)
Instrumental Constants	F	Estimated from design $F=60.00\pm0.10$ mm.	$(\sigma F/F)^2$	278				
	S	Direct measurement (Our instrument 122.24 ± 0.03 mm.)	$(\sigma S/S)^2$	6				
Physical Constants from the Literature	λ	It is estimated that the center of mass of a K_α doublet is known to 1/10 of the doublet separation $\sigma\lambda=4\times10^{-4}$ Å	$(\sigma\lambda/\lambda)^2$	7(Cu)		32(Mo)		32(Mo)
	λ_0		$(\sigma\lambda_0/\lambda_0)^2$			7(Cu)		7(Cu)
	A_0	Taken as twice the variation in spacing* of various recorded measurements of samples of natural quartz $(\sigma A_0/A_0=2\times10^{-4})$	$(\sigma A_0/A_0)^2$		4	4	4	4
Linear Measurements on Films	X	Statistical analysis of measurements on film (section 2)	$(\sigma X/X)^2$	>35	>35	>35	>9	>9
	X_0	Calibration repeated until mean error less than least count	$(\sigma X_0/X_0)^2$		35	35	9	9
	S_m	(See section 3)	$(\sigma S_m/S_m)^2$	35	35	35	9	9
	S_{m0}		$(\sigma S_{m0}/S_{m0})^2$		35	35	9	9
Calculated Spacing	A		$(\sigma A/A)^2\times10^8$	>361	>144	>183	>40	>79
			$(\sigma A/A)\times10^4$	>19	>12	>14	>6.3	>8.9

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* (Keith, 1950).

from several films on pages 330–331. We discuss the accuracy of angular measurements and consider the accuracy of the crystal cell dimensions in terms of the accuracy of the reciprocal net dimensions measured on the precession camera on page 331–332.

2. Reduction of Experimental Data and the Error in X and X_0

In the measuring device* used in the reduction of our data an engraved line on a plastic plate in contact with the film is constrained to move along, and is perpendicular to a scale on which the position of the line can be measured. With the film fixed in the measuring device and illuminated from below, the engraved line is set along each row of spots on the film and the coordinate x_r of each row with respect to the scale is recorded. The rows must, of course, be set accurately parallel to the engraved line before the measurements are made. In the instrument under discussion the vernier first used was calibrated to 0.1 mm. (least count 0.05 mm.) and was read to that accuracy. Columns 1–3 of Table 1 refer to the use of this vernier. In later measurements a vernier calibrated to 0.05 mm. (least count 0.025 mm.) was used. Columns 4 and 5 of Table 1 indicate the increase in accuracy resulting from the use of the more accurate device.

It is assumed that any departure of the set of measured values x_r from a uniform spacing of period X is due to experimental error and a least squares procedure is used to determine the best value of X and of the coordinate ξ of one row to fit the measured sequence of values x_r. If an odd number (2n+1) of rows** is measured as will almost always be the case on a zero layer photograph, it can easily be shown (cf. Whittaker and Robinson, 1924) that the best value of X is given by

$$X = [rx_r]/[r^2] \tag{4}$$

in which the bracket [ ] implies summation over all values of r from $-n$ to n. The corresponding values of the coordinate ξ of the central row is given by the average value.

$$\xi = [x_r]/(2n+1) \tag{5}$$

The standard deviations in X and ξ are respectively given by

$$(\sigma X)^2 = \Delta_0^2/\{(2n-1)[r^2]\} \tag{6}$$

and

$$(\sigma\xi)^2 = \Delta_0^2/(4n^2-1) \tag{7}$$

---

* Supplied by Charles E. Supper Co., Newton Centre, Mass.

** We have set up a similar discussion for an even number of rows but we have had no use for it and have not reported it here.

in which the sum of the squares of the residuals is given by

$$\begin{aligned}\Delta_0^2 &= [x_r^2] - \{[x_r]^2/(2n+1)\} - \{[rx_r]^2/[r^2]\} \\ &= [x_r^2] - \xi[x_r] - X[rx_r]\end{aligned} \tag{8}$$

It is useful to remember that

$$[r^2] = (1/3)n(n+1)(2n+1), \tag{9}$$

and it is easy to calculate a table of the values of this quantity to be expected from the crystals under investigation in a given laboratory.

In practice it is perhaps worthwhile to change the origin of x_r by subtracting the observed value of x_0 from the observed values of x_r thus minimizing the size of all the sums. In the usual zero layer measurements the value of ξ has no direct meaning. It is, however, worthwhile to calculate $\xi + rX$ for all lines. Comparison with the original values for X_r will indicate the presence of a mistake in the calculation or in the original measurements. An exact knowledge of ξ and its accuracy will, of course, be of importance in estimating the shift between the rows in the zero layer and those in a higher layer in monoclinic or triclinic crystals.

If a crystal has been set by standard routines so that only single spots are seen on the measured photographs, we believe that the quantity σX derived from (6) will include in itself all the errors which may arise from missetting, lack of centering, improper film positioning and kindred sources. For this reason we have made no other allowance for these possibilities in the present discussion.

It should be emphasized that the present reduction is not that which will produce the smallest value of σX from a given number of measurements. Repeated measurements of the more widely separated rows would lead to a lower standard deviation than does the present procedure. However, the latter gives a more realistic estimate of the reliability of the measurements made on the photograph.

It is clear that the least squares procedure can lead to ridiculous results, *i.e.*, to values of the standard deviation σX which are below the least count of the instrument. In such cases we have estimated the standard deviation of X by the formula

$$(\sigma X/X)^2 = 2M^2/L^2 \tag{10}$$

where L is the extreme range covered by the film (≈ 120 mm.) and M is the least count of the instrument. When $M = 0.05$ mm., $(\sigma X/X)^2 \approx 35 \times 10^{-8}$ and when $M = 0.025$ mm., $(\sigma X/X)^2 \approx 9 \times 10^{-8}$. These provide reasonable lower limits for the uncertainty in the measurement of X.

3. SHRINKAGE CORRECTIONS

In section 1 we have implied for simplicity that there is a pair of fiducial spots located so that the measurement of their separation S_m can be made at the angular setting of the measuring device used for the measurement of X. This is only true in the case of an orthogonal net set with one axis along the dial axis. The two fiducial spots along the dial axis will be measured at an angular setting ϕ_0 of the measuring device with the engraved line perpendicular to the line joining the spots. The average of the two measurements at ϕ_0 and $\phi_0+180°$ we call S_{mH}. The two fiducial spots perpendicular to the dial axis (Barnes *et al.*, 1951) are then measured at angular settings $\phi_0 \pm 90°$ with an average value which we call S_V. Assuming that the shrinkage is a homogeneous distortion with principal axes perpendicular to the edges of the film, the value of S_m to be used in correcting an X value measured at an angular setting ϕ will then be given by

$$S_m = \sqrt{S_H{}^2 \cos^2 (\phi - \phi_0) + S_V{}^2 \sin^2 (\phi - \phi_0)} \tag{11}$$

In Table 1 we have assumed that the average of two measurements for S_H or for S_V will be accurate to the least count of the measuring device *i.e.*, $(\sigma S_m/S_m)^2 = 2M^2/L^2$.

In measuring the angle between two rows on a film, two angles ϕ_1 and ϕ_2 must be measured. Each of these angles is subject to a small correction for shrinkage. The corrected value of ϕ_i is $\phi_i+\Delta$ where

$$\tan \Delta = -(S_H - S_V) \sin 2(\phi_i - \phi_0)/\{(S_H + S_V) + (S_H - S_V) \cos 2(\phi_i - \phi_0)\} \tag{12}$$

In most cases this correction will be of the same order of magnitude as the uncertainty in the measurement of the angle ϕ (Section 5). In all but the worst cases the approximate formula

$$\Delta = -\{(S_H - S_V)/(S_H + S_V)\} \sin 2(\phi_i - \phi_0) \tag{12a}$$

is quite accurate enough.

4. COMBINATION OF OBSERVATIONS FROM TWO OR MORE FILMS

If several different observations X_i corresponding to the same A are made on different films or on symmetrically equivalent rows on the same film, the results can be combined as follows. For a given film the ratio X_i/S_{mi} will have a weight w_i given by

$$1/w_i(X_i/S_{mi})^2 = \{(\sigma X_i/X_i)^2 + (\sigma S_{mi}/S_{mi})^2\} = 1/\omega_i \tag{13}$$

The most probable value of X/S_m will then be

$$X/S_m = \Sigma(w_i x_i/S_{mi})/\Sigma w_i \tag{14}$$

with a weight $W = \Sigma w_i$, where Σ implies summation over all observations. Thus we have

$$\{\sigma(X/S_m)/(X/S_m)\}^2 = 1/W(X/S_m)^2 = 1/\Omega \tag{15}$$

The actual calculation is facilitated by recognizing that $(X/S_m) \approx (X_i/S_{mi})$. Then with ω_i and Ω as defined by (13) and (15) respectively,

$$\Omega = \Sigma\omega_i \tag{16}$$

The lower limit of the relative standard deviation of X has already been given in (10), and in section 3 we have estimated $(\sigma S_m/S_m)^2$ as 2 $(M/L)^2$. Therefore it is clear that a minimum reasonable value for expression (15) is 4 $(M/L)^2$.

The discussion of sections 2–4 applies equally well to the measurement of X_0 and S_{m0} corresponding to the calibration spacing A_0. The number of films measured for calibration purposes should be sufficient to reduce the value of the expression (15) below the limit 4 $(M/L)^2$, and this has been assumed in Table 1, but it is not profitable to go beyond this number of measurements.

5. ERROR IN THE MEASUREMENT OF ANGLE

Two types of angular measurements can be made with the Buerger precession camera: (i) angles between nets, and (ii) angles between rows in a net.

Measurements of type (i) are made on the dial axis of the precession camera (calibrated to 5′) and depend for their accuracy on the precision with which a given net can be set parallel to the film. Experience shows that under the best conditions a missetting of the order of 0.2 mm. on the film can be detected, corresponding to about 3′ in angle. Thus allowing for a least count of 2.5′ the best accuracy to be achieved for a single angular setting will be about 4′. The measurement of angle will then be accurate to about 5′ or 6′. In any given experiment the investigator must estimate for himself the accuracy which he has been able to achieve in setting the two nets whose angular separation is to be measured. We have been unable to devise a reasonable method for the statistical analysis of this error.

Measurements of type (ii) are made on the film using the measuring device described above. This is provided with a circle calibrated to 5′. Independent measurements of the direction of a given family of rows may be made on the individual rows and also on these rows with the film holder rotated through 180°. The accuracy with which the device may be set parallel to a given set of rows depends very critically on the size of the crystal and the spots which it produces and also on the accuracy of the setting of the net. We have found standard deviations for a single setting as high as 17′ in the worst case we have met, but in the best cases measurements can be repeated to the least count. It is recommended that sufficient number of settings be made to establish a statistical estimate for the standard deviation of a single setting.

6. ERROR IN REDUCTION OF DATA FROM A SINGLE NET TO CELL DIMENSIONS

It must be noted that quantity A of Eqs (1)–(3) is a lattice translation a only if the two reciprocal nets which produce the rows in the precession photograph are perpendicular to the plane of the photograph. If they make an angle χ with this plane then

$$a = \frac{A}{\sin \chi} \tag{17}$$

and we must calculate the error in a from

$$(\sigma a/a)^2 = (\sigma A/A)^2 + (\sigma \chi)^2/\tan^2 \chi \tag{18}$$

in which the last term is negligible for angles near 90°. It may be too that the angle χ is not directly measured and that $\sin \chi$ must be calculated from other angles. In such cases the error analysis for the formula used must be carried out. Even though it may be necessary to use the exact formula for the calculation, the error analysis in terms of the exact formula may be cumbersome. In such cases the approximations for triclinic formulae discussed elsewhere prove useful (Patterson, 1952).

7. DISCUSSION

Examination of the column of Table 1 corresponding to equation (1) indicates that provided the manufacturing accuracy and adjustment is maintained a fractional accuracy of 20×10^{-4} can be expected in the measurement of a high quality film without calibration. This is to be compared with the figure of $20\text{-}30 \times 10^{-4}$ given by Evans (1949) and by Barnes and his coworkers (1951). When a careful calibration has been carried out and equation (2) or (3) can be used, errors respectively equal to 12 or 14×10^{-4} can be expected with the 0.10 mm. vernier and $6.3\text{-}8.9 \times 10^{-4}$ with the 0.05 mm. vernier. The contribution of the wave length error is much more important with the more accurate venier.

If all lattice translations are measured with approximately the same fractional accuracy, the contribution to the fractional error in a bond length by the errors in the lattice parameters will be approximately equal to the error in the lattice parameters. Thus a fractional error of 12×10^{-4} in these parameters would correspond to an error in a carbon single bond of about 0.002 Å so that cell parameters determined on the precession camera will in general be adequate for all but the most precise structure analyses.

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CRYSTAL CHEMISTRY AND SYSTEMATIC CLASSIFICATION OF HYDRATED BORATE MINERALS*

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Abstract

A systematic classification is made of hydrated borates of known crystal structures, based upon the structures of the complex boron-oxygen polyions contained in these crystals. From considerations of these known polyanion structures, four rules are deduced governing the nature of such polyanions. The structural formulas of a number of hydrated borates of known composition, but of unknown crystal structure, are predicted from the application of these rules and other reasonable crystal-chemical arguments.

Introduction

As a result of a number of investigations carried out in this and other laboratories in recent years, our knowledge of the crystal structures of hydrated borates has reached the stage where it is now feasible both to state several rules which seemingly govern the nature of the complex boron-oxygen polyions contained in such crystals, and to attempt a systematic classification of the known hydrated borates. It is the purpose of the present short paper to outline briefly some of the results obtained along these lines; a detailed paper is in preparation and will be published at some future time.

Four rules may be stated (Christ, 1959): (1) boron will link either three oxygens to form a triangle, or four oxygens to form a tetrahedron; (2) polynuclear anions are formed by corner sharing only of boron-oxygen triangles and tetrahedra in such a manner that a compact insular group of low to medium negative charge results; (3) in the polyions of hydrated borates, those oxygens not shared by two borons always attach a proton and exist as hydroxyl groups; (4) the insular groups may polymerize in various ways by splitting out water. As a corollary to the second rule it can be stated that the number of possible insular groups seems to be relatively small; as a corollary to the fourth rule, experience has shown that the insular groups can polymerize either to infinite chains, which may in turn be cross-linked, or possibly to dimers, but probably rarely or never to polymers of a degree between these two extremes.

In Table 1 are listed the polyions that have been definitely established by crystal-structure studies, the geometrical descriptions of these polyions, and the names, oxide formulas, and structural formulas of the compounds in which they are found. Illustrations have been omitted from the present paper; the reader is directed to the appropriate drawings in the original papers listed in the references.

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* Publication authorized by the Director, U. S. Geological Survey.

Table 1. Structural Formulas of Hydrated Borates of Known Crystal Structures

Name	Oxide Formula	Polyion	Description (Δ = triangle, t = tetrahedron)	Structural Formula	Investigator
Sassolite	$B_2O_3 \cdot 3H_2O$	$B(OH)_3$ (molecule)	Δ	$B(OH)_3$	Zachariasen (1954)
Teepleite Bandylite	$Na_2O \cdot B_2O_3 \cdot 2NaCl \cdot 4H_2O$ $CuO \cdot B_2O_3 \cdot CuCl_2 \cdot 4H_2O$	$[B(OH)_4]^{-1}$	t	$Na_2[B(OH)_4]Cl$ $Cu[B(OH)_4]Cl$	Fornaseri (1949) Collin (1951)
Pinnoite	$MgO \cdot B_2O_3 \cdot 3H_2O$	$[B_2O(OH)_6]^{-2}$	$2t$	$Mg[B_2O(OH)_6]$	Paton and MacDonald (1957)
Inyoite Synthetic Meyerhofferite	$2CaO \cdot 3B_2O_3 \cdot 13H_2O$ $2CaO \cdot 3B_2O_3 \cdot 9H_2O$ $2CaO \cdot 3B_2O_3 \cdot 7H_2O$	$[B_3O_3(OH)_5]^{-2}$	$2t$, 1Δ	$Ca[B_3O_3(OH)_5] \cdot 4H_2O$ $Ca[B_3O_3(OH)_5] \cdot 2H_2O$ $Ca[B_3O_3(OH)_5] \cdot H_2O$	Clark (1959) Clark and Christ (1959a) Christ and Clark (1956)
Colemanite	$2CaO \cdot 3B_2O_3 \cdot 5H_2O$	$[B_3O_4(OH)_3]_n^{-2n}$	$(2t, 1\Delta)_\infty$ chains	$Ca[B_3O_4(OH)_3] \cdot H_2O$	Christ *et al.* (1958)
Synthetic	$2CaO \cdot 3B_2O_3 \cdot H_2O$	$[B_3O_5(OH)]_n^{-2n}$	$(2t, 1\Delta)_{2\infty}$ sheets	$Ca[B_3O_5(OH)]$	Clark and Christ (to be published)
Monoclinic metaboric acid	$B_2O_3 \cdot H_2O$	$[B_3O_4(OH)_2]_n^{-n}$	$(1t, 2\Delta)_\infty$ chains	$H[B_3O_4(OH)_2]$	Zachariasen (1952)
Borax	$Ba_2O \cdot 2B_2O_3 \cdot 10H_2O$	$[B_4O_5(OH)_4]^{-2}$	$2t$, 2Δ	$Na_2[B_4O_5(OH)_4] \cdot 8H_2O$	Morimoto (1956)
"Potassium pentaborate tetrahydrate"	$K_2O \cdot 5B_2O_3 \cdot 8H_2O$	$[B_5O_6(OH)_4]^{-1}$	$1t$, 4Δ	$K[B_5O_6(OH)_4] \cdot 2H_2O$	Zachariasen (1938)

It is a reasonable extrapolation to extend these known structural formulas to those compounds of the same stoichiometric composition. This has been done in Table 2. Thus, it might be expected that inderite, $2MgO \cdot 3B_2O_3 \cdot 15H_2O$, would contain the same $[B_3O_3(OH)_5]^{-2}$ rings found in inyoite, or, less likely, the chains of composition $[B_3O_4(OH)_3]_n^{-2n}$ found in colemanite. The structural formula assigned to a compound by extrapolation in this way will depend upon the degree of polymerization assumed. In every case the polymerization involves the splitting out of water molecules between two or more insular groups. Any specific case can be understood in terms of the example afforded by the schematic relationship between meyerhofferite and colemanite which may be written:

$$\underset{\text{meyerhofferite}}{n[B_3O_3(OH)_5]^{-2}} = \underset{\text{colemanite}}{[B_3O_4(OH)_3]_n^{-2n}} + nH_2O$$

Table 2 lists the probable structural formulas for a number of compounds whose crystal structures are unknown at present. Where there is no evidence to the contrary it is assumed that the polyions are monomeric; if there are data leading to a definite polymer, this polymer is the one listed. Space group and unit-cell volume data are important in limiting the possible polymers, and these have been taken into account in the preparation of Table 2 but are not considered explicitly there.

In the first part of Table 2 are given the structural formulas directly derivable from well-known polyion types. Thus, the formula of frolovite may be derived from that of teepleite or that of pinnoite (Table 1). Inderite, kurnakovite, inderborite, and hydroboracite all have a 2:3:x oxide ratio and are presumed to be related to the known inyoite-colemanite series. Tincalconite probably contains the same polyions as borax, while kernite probably contains infinite chains resulting from the polymerization of these polyions (Christ and Garrels, 1959). A synthetic compound of composition $2CaO \cdot 3B_2O_3 \cdot H_2O$ (Table 1), prepared by Brian J. Skinner, U. S. Geological Survey, by heating inyoite with water at 400° C. and 2000 bars for several days, has been shown to contain sheets consisting of cross-linked colemanite-type chains and has the structural formula $CaB_3O_5(OH)$ (Clark and Christ, to be published). For hilgardite (Hurlbut and Taylor, 1937) and parahilgardite (Hurlbut, 1938) the analyses of Gonyer lead to the composition $2CaCl_2 \cdot 6CaO \cdot 9B_2O_3 \cdot 4H_2O$. This composition is explained (Table 2) if we assume that these two minerals contain the same kind of sheets found in the synthetic compound $CaB_3O_5(OH)$.*

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* Braitsch (1959a), as a result of new analyses, proposes the formula $Ca_2B_5O_8(OH)_2Cl$ for parahilgardite and hilgardite, and $(Ca, Sr)_2B_5O_8(OH)_2Cl$ for a new strontian hilgardite found by him. This work still needs assessment.

TABLE 2. POSTULATED STRUCTURAL FORMULAS OF HYDRATED BORATES OF UNKNOWN CRYSTAL STRUCTURES

Name	Oxide Formula	Polyion	Description (Δ=triangle, t=tetrahydron	Structural Formula	References* and Remarks
Frolovite	$CaO \cdot B_2O_3 \cdot 4(?)H_2O$	$[B(OH)_4]^{-1}$ *or* $[B_2O(OH)_6]^{-2}$	*t or 2t*	$Ca[B(OH)_4]_2$ *or* $Ca[B_2O(OH)_6] \cdot H_2O$	Petrova (1957) (Petrova gives $3\frac{1}{2}H_2O$ in oxide formula)
Inderite Kurnakovite Inderborite	$2MgO \cdot 3B_2O_3 \cdot 15H_2O$ $2MgO \cdot 3B_2O_3 \cdot 15H_2O$** $MgO \cdot CaO \cdot 3B_2O_3 \cdot 11H_2O$	$[B_3O_3(OH)_5]^{-2}$	$2t$, 1Δ	$Mg[B_3O_3(OH)_5] \cdot 5H_2O$ $Mg[B_3O_3(OH)_5] \cdot 5H_2O$ $MgCa[B_3O_3(OH)_5]_2 \cdot 6H_2O$	Vol. 2, p. 360 Vol. 2, p. 360 Vol. 2, p. 355
Hydroboracite	$MgO \cdot CaO \cdot 3B_2O_3 \cdot 6H_2O$	$[B_3O_4(OH)_3]_n^{-2n}$	$(2t, 1\Delta)_\infty$ chains	$MgCa[B_3O_4(OH)_3]_2 \cdot 3H_2O$	Vol. 2, p. 353
Tincalconite	$Na_2O \cdot 2B_2O_3 \cdot 5H_2O$	$[B_4O_5(OH)_4]^{-2}$	$2t$, 2Δ	$Na_2[B_4O_5(OH)_4] \cdot 3H_2O$	Vol. 2, p. 337 Christ and Garrels (1959)
Kernite	$Na_2O \cdot 2B_2O_3 \cdot 4H_2O$	$[B_4O_6(OH)_2]_n^{-2n}$	$(2t, 2\Delta)_\infty$ chains	$Na_2[B_4O_6(OH)_2] \cdot 3H_2O$	Vol. 2, p. 335 Christ and Garrels (1959)
Hilgardite *or* parahilgardite	$2CaCl_2 \cdot 6CaO \cdot 9B_2O_3 \cdot 4H_2O$	$[B_3O_5(OH)_n^{-2n}$	$(2t, 1\Delta)_{2\infty}$ sheets	$Ca_8[B_3O_5(OH)]_6Cl_4 \cdot H_2O$	Vol. 2, pp. 382–383
Gowerite	$CaO \cdot 3B_2O_3 \cdot 5H_2O$	$[B_3O_3(OH)_4]^{1}$	$1t$, 2Δ	$Ca[B_3O_3(OH)_4]_2 \cdot H_2O$	Erd *et al.* (1959) Christ and Clark (1960)
Veatchite	$SrO \cdot 3B_2O_3 \cdot 2(?)H_2O$	$[B_6O_7(OH)_6]^{-2}$	$(1t, 2\Delta)_2$ dimers	$Sr[B_6O_7(OH)_6]$	Clark *et al.* (1959, 1960) This formulation requires at least $3H_2O$ in oxide formula.
p Veatchite	$SrO \cdot 3B_2O_3 \cdot 2(?)H_2O$	$[B_6O_7(OH)_6]^{-2}$	$(1t, 2\Delta)_2$ dimers	$Sr[B_6O_7(OH)_6]$	Braitsch (1959b) Remarks above apply here also.
Ammonioborite Larderellite Sborgite	$(NH_4)_2O \cdot 5B_2O_3 \cdot 5\frac{1}{3}H_2O$ $(NH_4)_2O \cdot 5B_2O_3 \cdot 5(?)H_2O$ $Na_2O \cdot 5B_2O_3 \cdot 10H_2O$	$[B_5O_6(OH)_4]^{-1}$	$1t$, 4Δ	$NH_4[B_5O_6(OH)_4] \cdot \frac{2}{3}H_2O$ $NH_4(B_5O_6(OH)_4] \cdot \frac{1}{2}(?)H_2O$ $Na[B_5O_6(OH)_4] \cdot 3H_2O$	Clark and Christ (1959b) Clark and Christ (1959b) Cipriani (1957)
Ezcurrite	$2Na_2O \cdot 5B_2O_3 \cdot 7H_2O$	$[B_5O_6(OH)_5]^{-2}$	$2t$, 3Δ	$Na_2[B_5O_6(OH)_5] \cdot H_2O$	Muessig and Allen (1957)
Ulexite Probertite	$Na_2O \cdot 2CaO \cdot 5B_2O_3 \cdot 16H_2O$ $Na_2O \cdot 2CaO \cdot 5B_2O_3 \cdot 10H_2O$	$[B_5O_6(OH)_6]^{-3}$	$3t$, 2Δ	$NaCa[B_5O_6(OH)_6] \cdot 5H_2O$ $NaCa[B_5O_6(OH)_6] \cdot 2H_2O$	Vol. 2, p. 345 Clark and Christ (1959c) Vol. 2, p. 343 Clark and Christ (1959c)
Preobrazhenskite	$3MgO \cdot 5B_2O_3 \cdot 4\frac{1}{2}H_2O$	$[B_5O_7(OH)_4]_n^{-3n}$	$(3t, 2\Delta)_\infty$ chains	$Mg_3[B_5O_7(OH)_4]_2 \cdot \frac{1}{2}H_2O$	Yarzhemskii (1956)
Tertschite	$4CaO \cdot 5B_2O_3 \cdot 20H_2O$	$[B_5O_6(OH)_7]^{-4}$	$4t$, 1Δ	$Ca_2[B_5O_6(OH)_7] \cdot 6\frac{1}{2}H_2O$	Meixner (1953)
Kaliborite	$K_2O \cdot 4MgO \cdot 11B_2O_3 \cdot 18H_2O$	$[B_5O_6(OH)_4]^{-1}$ *and* $[B_3O_3(OH)_5]^{-2}$	$1t$, 4Δ *and* $2t$, 1Δ	$KMg_2[B_5O_6(OH)_4][B_3O_3(OH)_5]_2 \cdot 2H_2O$	Vol. 2, p. 367

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* Vol. 2 in this column stands for Palache, Berman, and Frondel (1951).

** Oxide formula with 15 H_2O is correct. (Schaller and Mrose, private communication.)

Zachariasen (1952) has shown that crystalline monoclinic metaboric acid, HBO_2, contains chains of composition $[B_3O_4(OH)_2]_n^{-n}$. The corresponding insular ring, consisting of two triangles and a tetrahedron, would have the formula $[B_3O_3(OH)_4]^{-1}$. Either the chains or the rings (or intermediate polymers) would explain minerals of the type $MO \cdot 3B_2O_3 \cdot xH_2O$, such as gowerite (Erd *et al.*, 1959; Christ and Clark, 1960), veatchite (Clark *et al.*, 1959; Clark and Mrose, 1960), or *p*-veatchite (Braitsch 1959b), as shown in Table 2. For veatchite, $SrO \cdot 3B_2O_3 \cdot 2(?)H_2O$, preliminary crystal structure studies (Clark and Christ, to be published) indicate the presence of dimers of composition $[B_6O_7(OH)_6]^{-2}$. This assumption leads to the structural formula $SrB_6O_7(OH)_6$, and the corresponding oxide formula $SrO \cdot 3B_2O_3 \cdot 3H_2O$. Presumably *p*-veatchite also contains these dimers.

The remaining polynuclear ion to be considered is the pentaborate ion discovered by Zachariasen (1938) in "potassium pentaborate tetrahydrate," $K_2O \cdot 5B_2O_3 \cdot 8H_2O$. Zachariasen formulated this pentaborate ion as $[B_5O_8(OH)_2]^{-3}$, assumed that the crystal contained hydronium ions, and wrote the formula of the compound as $KH_2(H_3O)_2B_5O_{10}$. However, since aqueous solutions of all of the known borates are weakly to strongly alkaline (except for the weak acid orthoboric acid) it seems very unlikely that in this or any other hydrated borate crystal will the polyion act as a proton donor. The formation of hydronium ions in crystals appears to take place only in the hydrates of strong acids, as for example in $HNO_3 \cdot 3H_2O$ (Luzzati, 1953). This conclusion is in agreement with the results of proton magnetic resonance studies of Silvidi and McGrath (1959) and Smith and Richards (1952). With these considerations in mind and invoking Rule 3, we accept the main results of Zachariasen and conclude that potassium pentaborate tetrahydrate contains polyions of composition $[B_5O_6(OH)_4]^{-1}$, consisting of one tetrahedron and four triangles linked at corners and assign to the compound the structural formula $K[B_5O_6(OH)_4] \cdot 2H_2O$.* The structural formulas for the minerals larderellite and ammonioborite (Table 2) follow immediately from these considerations.

On purely stereochemical grounds one might expect to be able to replace each of the $O_2B(OH)$ triangles of the pentaborate ring successively by $O_2B(OH)_2$ tetrahedra. Such a procedure will lead from the $1{:}5{:}x$ ratio of potassium pentaborate tetrahydrate to the successive ratios

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* The results of Silvidi and McGrath (1959) also indicate to them that the pentaborate ring contains an *intra* hydrogen bond (Fig. 4, their paper). The fact that such a bond would place the proton at about 1 Å from the highly electropositive boron renders this postulate completely untenable. The stoichiometry of the compound indicated by their Fig. 4 is also puzzling.

2:5:x, 3:5:x, 4:5:x, and 5:5:x. This has been done in postulating structural formulas in Table 2 for ezcurrite, 2:5:7, ulexite, 3:5:16, probertite, 3:5:10, preobrazhenskite, 3:5:4½, and tertschite, 4:5:20. Presumably, minerals of ratio 5:5:x, containing pentaborate ions consisting of five linked tetrahedra would be unstable toward minerals of the pinnoite structure which contains polyions of two linked tetrahedra (Table 1).

Finally, in Table 2 we list kaliborite which probably represents the unusual case of a mineral containing two distinct kinds of polyions.

A large number of synthetic hydrated borates have been reported in the literature and are summarized in Kemp (1956). In the interests of brevity these have not been considered here, but will be taken up in the more detailed paper.

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CLINOPTILOLITE FROM PATAGONIA THE RELATIONSHIP BETWEEN CLINOPTILOLITE AND HEULANDITE

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ABSTRACT

Clinoptilolite occurs as a layer 6–8 cm. thick interbedded with bentonitic clays of the Sarmiento formation (Eocene-Oligocene) in the Cañadón Hondo in Argentine Patagonia. Physical and optical data are given for the mineral; its density is 2.11, mean refractive index 1.478, birefringence about 0.001. A chemical analysis indicates a unit cell content of $(Na, K)_{4.86}(Ca, Mg)_{0.31}Al_{5.49}Si_{30.50}O_{72}\cdot 19H_2O$. The mineral has been formed by diagenetic alteration of volcanic glass in a non-marine environment.

Comparison of this material with other clinoptilolites, including the original material described by Pirsson (1890) and named by Schaller (1923), and with heulandite, shows that clinoptilolite is a distinct species. Clinoptilolite and heulandite have the same structure, but heulandite is essentially a calcium zeolite and clinoptilolite a sodium-potassium zeolite; analyses of the minerals indicate that there is a compositional gap between them.

INTRODUCTION

The material described in this paper was collected by Dr. G. G. Simpson in February 1931 during the Scarritt Patagonian Expedition of the American Museum of Natural History. Descriptions of some of the rock specimens were published by McCartney (1933), but he did not include any from the Cañadón Hondo. His paper, however, contains a sketch map showing the location of the Cañadón Hondo, which is a tributary of the Rio Chico some 40 miles northwest of Comodoro Rivadavia, in Argentine Patagonia.

A description of the geology in the Cañadón Hondo had not been published, but Dr. Simpson has permitted us to quote the following from his field notes:

> "Cañadón Hondo is remarkably confusing and no clear idea of its geology can be obtained short of a detailed survey. Its great size, very complex and obscure structure with numerous faults and folds, the discontinuity of exposures or structures at critical points, the lenticular nature of the beds, the strangeness of their facies, and the general paucity of fossils make any very definite conclusions impossible.
>
> Aside from the Patagoniano (late Oligocene-early Miocene) which is always readily recognizable and usually confined to the higher rim, the following types of sediments occur:
>
> 1. More or less massive white, pink, or rarely yellowish tuffs, with some tuffaceous clay.
> 2. Pale green, occasionally highly silicified and often very concretionary tuffs intercalated with bright green clay.
> 3. Green clay with spots and poorly sorted pebbles, associated with bright green sands and, rarely white sands.
> 4. Pale clays, massive, grey, pinkish, greenish, and fine to coarse crossbedded sands,

> usually white or grey, but often greenish or pink to red. Although physically similar and not separable at present, these include two parts probably quite distinct in age:
>
> *a.* An upper series in which the pillar bed and mammal sandstone are intercalated.
>
> *b.* A lower series including true Banco negro.
>
> In (2), probably in its lower part only, are also seen two very special types: thin widespread white beds supposed in the field to be diatomaceous earth, and finely laminated shales with local plants and fish."

From subsequent study, Dr. Simpson now adds the following observations:

> "At least 1–2 of the above lithological units, and perhaps also 3, are unusual rock facies within the predominantly volcanic Eocene-Oligocene mass of sediments now sometimes lumped as the Sarmiento group or (less correctly) formation. Casamayoran fossils were found in beds included in facies 2 of the field notes, and that is the probable age of the clinoptilolite. 4a is definitely older and was later made the type of the Rio Chico formation and Riochican age and stage. The dating of these provincial ages in terms of the world scale is quite uncertain, but most vertebrate paleontologists now consider the Riochican as approximately late Paleocene and the Casamayoran as approximately early Eocene.
>
> In 1–3, at least, the 'clay' of the field notes is bentonite. All these beds are continental. They are bounded above by the marine Patagonia formation ('Patagoniano') of latest Oligocene or early Miocene age and below by the marine Salamanca formation, probably of early Paleocene age."

In the last sentence of the quotation from the field notes there is a reference to thin widespread white beds supposed in the field to be diatomaceous earth. In 1956 a chance-taken *x*-ray powder photograph of one of these samples revealed that it was a zeolite with the heulandite structure, subsequently identified as clinoptilolite.

General Description

The material available for investigation consisted of two specimens, both evidently the full thickness of the clinoptilolite band, since they were bounded on two surfaces by bedding planes with adherent clay. One had a thickness of 6 cm. and the other of 8 cm. Since the thicker specimen was also the purer and more homogeneous, it was investigated in detail.

One bedding surface of the specimen was coated with a layer, 1 mm. thick, of translucent barite, to which was adhering some fragments of dense pale green bentonitic clay. The thin layer of barite was followed by 6 cm. of white chalky clinoptilolite, quite tough, with a conchoidal fracture. The succeeding 2 cm. showed streaks of pale green bentonitic clay interlayered with the clinoptilolite, and the clinoptilolite showed a granular instead of a dense compact structure. A thin section cut across this part of the specimen shows that the main part of the specimen (the dense compact material) is pure clinoptilolite, apparently formed by the alteration of fine-grained (less than 20 microns) volcanic glass; it has rare thin veinlets of opal and montmorillonite. The granular material is a mixture of orthoclase and clinoptilolite, and the bentonitic clay consists essentially of montmorillonite.

A specimen of the clinoptilolite was analyzed in more detail by making size separations and studying the size fractions in order to detect small amounts of other phases. *X*-ray analysis of the size fractions showed a small amount of montmorillonite and opal (alpha-cristobalite) in the -8 micron size fractions. In thin section, the opal and montmorillonite were seen to be restricted to a few thin veins (less than 0.3 mm. thick) cutting the clinoptilolite and extending from the bordering montmorillonite. Unaltered volcanic glass was found in small amount ($<1\%$) only in the coarsest size fraction.

Table 1. *X*-Ray Powder Diffraction Data on Clinoptilolite from Patagonia
(Relative intensities visually estimated)

d (Å)	I	d(Å)	I	d(Å)	I	d(Å)	I
8.96	vs	3.55	w	2.54	w	1.701	vw
7.94	m	3.43	m	2.44	w	1.664	vw
6.80	w	3.33	vw	2.36	vw	1.597	vw
5.98	vw	3.18	w	2.19	vw	1.523	vw
5.13	m	3.10	vw	2.09	vw	1.488	vw
4.65	w	2.98	s	2.02	w	1.416	vw
4.35	w	2.81	m	1.955	w	1.350	vw
3.97	vs	2.74	w	1.829	vw	1.315	vw
3.72	w	2.68	vw	1.776	vw		

In immersion liquids under the microscope crushed fragments of the clinoptilolite show no sign of crystal form. The granules have a pale brownish turbidity. The mean refractive index is 1.478 and the birefringence very low, about 0.001.

An electron micrograph of dispersed clinoptilolite shows that part at least is present as well-formed plates or laths; these plates evidently reflect the development of the form (010), a prominent form and the single cleavage in heulandite. Because of the very small particle size, and the fact that the clinoptilolite is itself nearly isotropic, the texture of a fracture surface was examined in the electron microscope by the preshadowed carbon replica technique. The surface appeared very homogeneous and exhibited an irregular fracture pattern. No smooth areas with conchoidal fracture which would indicate the presence of unaltered glass were found.

An *x*-ray powder photograph was made of a -200 mesh size fraction of the clinoptilolite, and the d-spacings and intensities of the lines are given in Table 1. The powder photograph shows that the material is a well-crystallized clinoptilolite.

Coombs (1958) has compared powder photographs of clinoptilolite and heulandite and points out two main types of difference between them: (a) a tendency towards larger spacings in clinoptilolite, which he ascribes to the effect of the large potassium ion involved in the replacement of CaAl in heulandite by (Na, K) Si in clinoptilolite; (b) differences in relative intensities, resulting from the method of mounting combined with differences in crystallinity of the samples. The strong (020) reflection at about 9 Å is enhanced by preferred orientation of (010) cleavage flakes in macroscopically crystallized material.

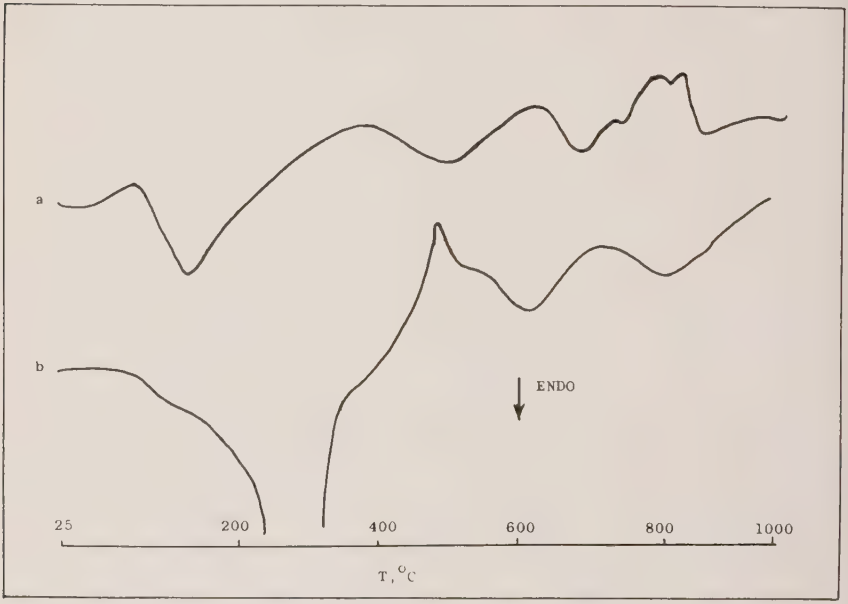

FIG. 1. Differential thermograms of *a*) clinoptilolite, Patagonia; *b*) heulandite, Paterson, New Jersey.

The differential thermal analysis curves for the clinoptilolite and for a heulandite are given in Fig. 1. The differences are marked and characteristic. Mumpton (1958) states that at about 230° C. heulandite transforms to "heulandite B" and at 350° C. becomes amorphous, whereas clinoptilolite remains stable to about 700° C. without reaction at lower temperatures. However, the endothermic reaction just below 200° C. on our D.T.A. curve for clinoptilolite presumably reflects the loss of at least part of the constitutional water.

The determination of the true density of the clinoptilolite presents some problems, on account of the fine-grained and porous nature of the material. A number of small fragments were broken from the purest part of the specimen, placed in a glass cylinder, and covered with a bell jar. The bell jar was thoroughly pumped out with a vacuum pump, and a mixture of methylene iodide and acetone ($D = 2.20$) was then run into the cylinder with the fragments. All the fragments floated. Acetone was added in small amounts until the fragments were in suspended equilibrium, and the density of the liquid then determined. In this way the density of the clinoptilolite was found to be 2.11 ± 0.02.

Origin of the Clinoptilolite

The occurrence of this thin bed of clinoptilolite in a thick sequence of bentonitic clays presents an interesting problem of origin. The geological evidence indicates that both the clinoptilolite bed and the bentonitic clays are the products of diagenesis of volcanic glass tuffs laid down in a non-marine environment. Two hypotheses, not mutually exclusive, may be suggested:

(1) The clinoptilolite bed was formed from a volcanic glass of composition rather similar to that of the clinoptilolite, and different from the volcanic glass from which the bentonitic clays were derived.

(2) The initial composition of the volcanic glass was similar throughout, but special conditions of diagenesis led to the formation of the clinoptilolite.

For the first hypothesis, a rhyolitic glass practically lacking in iron, magnesium, and calcium would be a satisfactory parent material for the clinoptilolite, whereas the bentonitic clays would be derived from material of dacitic or andesitic composition. This hypothesis is supported by the occurrence, at another locality in the Cañadón Hondo, of a thin white chalk-like layer interbedded with bentonitic clays. This white layer was identified on sight as clinoptilolite also, but proved to consist of colorless volcanic glass containing numerous small orthoclase crystals—the type of parent material postulated above for clinoptilolite.

The second hypothesis envisages a difference in composition and circulation of the diagenetic waters acting on volcanic glass of fairly uniform composition. Clinoptilolite would be formed when the volcanic glass was acted on by waters rich in sodium and potassium -possibly an alkaline lake water similar to those of the alkaline lakes of Nevada and California. Montmorillonite would form where circulating waters removed some silica and most of the alkalies. This hypothesis suggests that the clinoptilolite formed during a short-lived episode when the volcanic glass was being deposited in an evanescent alkaline lake.

We have discussed these hypotheses with Dr. Simpson, who makes the following comment:

> "I see no objection to either hypothesis, and I know nothing that would really help to choose between them.

TABLE 2. CHEMICAL ANALYSIS OF CLINOPTILOLITE FROM PATAGONIA
ANALYST: H. B. WIIK

	1	2		3	4
SiO_2	69.07	69.41	Si	30.91	30.50
TiO_2	0.04	0.04	Al	5.57	5.49
Al_2O_3	10.88	10.59	Mg	0.13	0.12
Fe_2O_3	0.08	0.08	Ca	0.20	0.19
FeO	0.00	0.00	Na	3.83	3.78
MnO	0.01	0.01	K	1.10	1.08
MgO	0.18	0.19	$H_2O\pm$	19.27	19.01
CaO	0.39	0.41	O	72.96	72.00
Na_2O	4.23	4.42			
K_2O	2.52	1.92			
P_2O_5	0.00	0.00			
$H_2O+110°$	6.56	6.85			
$H_2O-110°$	5.82	6.08			
CO_2	0.00	0.00			
	99.78	100.00			

1. Weight per cent
2. Recalculated to 100, after deducting 4% $KAlSi_3O_8$.
3. Atoms per unit cell.
4. Atoms per unit cell, recalculated on the basis of $O=72$.

Simply on speculative weighing of possibilities, (1) seems to me somewhat more likely. One can easily imagine that a nearly unique weather event (some very unusual air movement) could once or twice carry a thin deposit of ash from a distant rhyolitic source into a momentarily quiescent area of long-continued dacitic or andesitic vulcanism. It seems (still speculatively) a little less likely that formation of an alkaline lake would be so nearly unique. If the climatic conditions were right, one would expect that other such deposits would occur in so thick a series over so large an area, through so long a time. Lake deposits are known elsewhere in Patagonia during this time, and in them the ash is bentonitic. The faunas suggest a fairly warm to humid climate throughout this time. The floras are very poorly known but are at least consistent with the humid climate. So this one alkaline lake would be extremely exceptional, but of course not impossible."

CHEMICAL COMPOSITION

A sample of the clinoptilolite was taken for chemical analysis from the purest middle section of the band, and was selected to avoid any of the small veinlets of opal in the material. When crushed and examined in immersion mounts under the microscope the only foreign material seen was occasional grains of orthoclase; a grain count indicated an amount of 4%.

The sample was analyzed by Dr. H. B. Wiik. The results are given in Table 2.

In order to correct for the presence of the small amount of orthoclase, amounts of K_2O, Al_2O_3, and SiO_2 corresponding to 4% $KAlSi_3O_8$ have been deducted from the analysis figures, and the analysis then recalculated to 100. Since the orthoclase probably contains some sodium replacing potassium, this procedure results in a distortion of the relative amounts of sodium and potassium in the clinoptilolite. However, the distortion is certainly small, and does not affect the total molecular proportion of sodium plus potassium.

Hey and Bannister (1934) have shown that the unit cell formula for heulandite (and clinoptilolite) is $Na_xCa_yAl_{x+2y}Si_{36-(x+2y)}O_{72}\cdot 24H_2O$, where $(x+y)$, the number of large cations per unit cell, is variable, ranging from 4 to 6. The corrected analysis of the Patagonia clinoptilolite (omitting those components present in amounts less than 0.1%) has been recalculated into atoms per unit cell, using the cell dimensions $a=7.46$, $b=17.84$, $c=15.88$, $\beta=91°26'$ given for heulandite by Strunz and Tennyson (1956), and the measured density of 2.11. When this is done the oxygen content is 72.96, slightly higher than the true value of 72. On recalculation to $O=72$, $Si+Al$ becomes 35.99, very close to the true value of 36; the total $Ca+Mg+Na+K$ is 5.17; $H_2O^\pm$ is 19.01, instead of the ideal value of 24. The low figure for water in the Patagonia clinoptilolite is probably due to its having been stored in a warm dry museum for twenty-seven years prior to analysis, with the consequent loss of weakly bonded water.

The Relationship Between Clinoptilolite and Heulandite

Pirsson (1890) described as mordenite a crystalline mineral, isomorphous with heulandite, occurring in a highly weathered amygdaloidal basalt in the Hoodoo Mountain, Wyoming. Schaller (1923) recognized that Pirsson's mineral was not mordenite and regarded it as an independent species, naming it clinoptilolite. Hey and Bannister (1934) studied Pirsson's original material by *x*-rays, and could find no differences between it and heulandite. Chemical analyses of clinoptilolite and heulandite could be expressed by the same unit cell formula, given above. They pointed out that analyses of clinoptilolite were higher in SiO_2 and lower in Al_2O_3 than those of heulandite, and concluded that clinoptilolite . . . "are essentially high-silica heulandites . . . the name clinoptilolite is unsuitable and should not be used." Mumpton (1958) claims that the base:alumina:silica ratio for heulandite is 1:1:6 and for clinoptilolite 1:1:8, that no specimens of these minerals have been found with compositions or properties intermediate between the two end members, and therefore proposes to redefine clinoptilolite as the high-silica counterpart

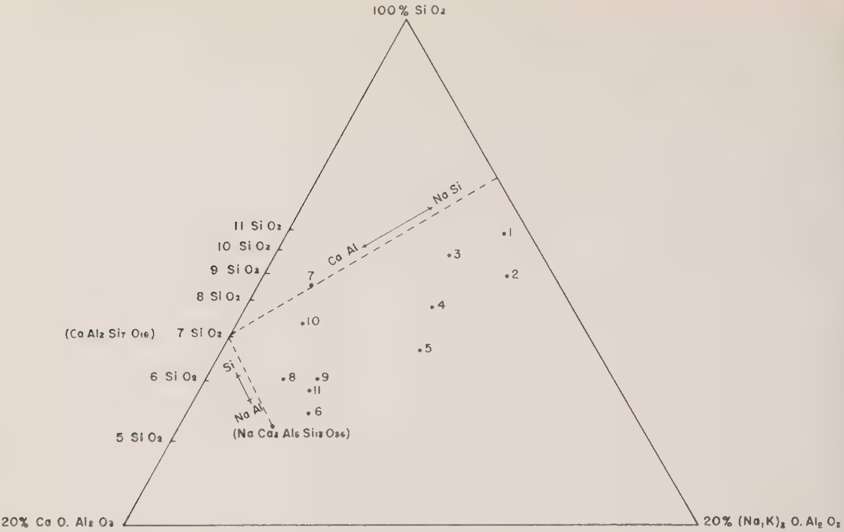

FIG. 2. Analyses of clinoptilolite and heulandite plotted in molecular percentages of the components $CaO \cdot Al_2O_3$, $(Na, K)_2O \cdot Al_2O_3$ and SiO_2. The dashed lines show the changes in composition produced by the substitution of NaAl for Si, and of NaSi for CaAl, in the ideal formula for heulandite, $CaAl_2Si_7O_{18}$.

1. Clinoptilolite, Patagonia. $n = 1.478$, birefringence 0.001.
2. Clinoptilolite, Hector, California. *Econ. Geol.* **53,** 31 (1958), $n = 1.480$.
3. Clinoptilolite, Hoodoo Mt., Wyoming. *Am. Jour. Sci.* **40,** 235 (1890), $\alpha = 1.476$, $\gamma = 1.479$.
4. Clinoptilolite, Dome, Arizona. *Am. Mineral.* **18,** 170 (1933).
5. Clinoptilolite, Challis, Idaho. *Proc. U. S. Nat. Mus.* **64,** Art. 19, 12 (1924), (described as heulandite). $\alpha = 1.482$, $\beta = 1.485$, $\gamma = 1.489$.
6. Heulandite, Hawaii. *Am. Mineral.* **18,** 369 (1933), $\alpha = 1.501$, $\beta = 1.504$, $\gamma = 1.509$.
7. Heulandite, Croatia. *Zentr. Mineral.*, Abt. A, 227 (1940), $\beta = 1.488$, birefringence 0.001.
8. Heulandite, Italy. *Rec. Accad. Sci. Torino* **64,** 105 (1929), $\beta = 1.500$.
9. Heulandite, Sardinia. *Re. Accad. Lincei* **18,** 80 (1909).
10. Heulandite, Japan. *Zeits. Krist.* **52,** 513 (1913).
11. Heulandite, Iceland. *Zeits. Krist.* **49,** 201 (1911).

of heulandite and to retain the name as a valid mineral species, on equal status with heulandite.

When, however, analyses of heulandite and clinoptilolite are examined, it appears that the differences lie not so much in the silica content, as Hey and Bannister and Mumpton maintain, but in the content of sodium and potassium. The compositional relationships and differences are illustrated in Fig. 2, in which analyses of clinoptilolite are plotted in terms of molecular percentages of the components SiO_2, $CaO \cdot Al_2O_3$, and $(Na,K)_2O \cdot Al_2O_3$. Since all the analyses when recalculated in this way contain more than 80% SiO_2 and less than 20% of the other components, only this part of the three-component diagram SiO_2–$CaO \cdot Al_2O_3$–$(Na,K)_2O \cdot Al_2O_3$ is used in Fig. 2.

On Fig. 2 are also plotted six analyses of heulandite, selected as showing the highest values recorded for Na_2O and K_2O, and the greatest variation in SiO_2 content. The figure reveals a definite composition gap between heulandite and clinoptilolite, suggesting that although these two minerals have similar structures, a complete solid solution series between them does not occur.

We therefore believe that clinoptilolite is a distinct species, and should be defined as a zeolite with the heulandite structure in which (Na+K) in atoms per unit cell is predominant over Ca. The type material designated by Schaller, i.e., that described by Pirsson from Hoodoo Mountain, Wyoming, agrees with this definition.

Analysis 5 on Fig. 2 is particularly significant for discriminating between clinoptilolite and heulandite. Originally described as heulandite, it has a alumina:silica ratio of 1:6.7, and would therefore be classified by Mumpton as a heulandite. However, its molecular content of Na+K exceeds that of Ca+Mg, and we consider it to be a clinoptilolite. We have obtained a sample of the original material described by Ross and Shannan through the courtesy of the U. S. National Museum. The differential thermogram corresponds to that of clinoptilolite. When tested as suggested by Mumpton (heated in an oven at 450° C. overnight and an x-ray powder photograph then run) it also behaved as a clinoptilolite, viz. a good powder photograph was obtained.

Wyart (1933), in a comprehensive study of heulandite, concluded that the ideal formula of this mineral is $CaAl_2Si_7O_{18} \cdot 6H_2O$, there being four of these formula units in the unit cell. He studied some twenty-six analyses of heulandite, and showed that the compositional variations could be largely explained by a replacement of Si by NaAl; natural heulandites he regarded as solid solutions in various proportions of the two components $Ca_2Al_4Si_{14}O_{36} \cdot 12H_2O$ and $NaCa_2Al_5Si_{13}O_{36} \cdot 12H_2O$. However, study of Fig. 2 shows that this is an over-simplification. Lines are drawn to indicate the variations in composition that can be produced by ionic substitutions of the type $NaAl \leftrightarrow Si$ and $NaSi \leftrightarrow CaAl$ in the ideal formula for heulandite. In terms of ionic substitution clinoptilolite bears a similar relationship to heulandite as albite does to anorthite.

On the $CaO \cdot Al_2O_3$—SiO_2 axis of Fig. 2 the points corresponding to integral values of x in $CaO \cdot Al_2O_3 : xSiO_2$ from 5 to 11 have been indicated. The purpose of this is to show that formulas written in this fashion are unsuitable for representing the compositions of these zeolites. In the analyses of the heulandites the molecular ratio of SiO_2, to Al_2O_3 ranges from about $5\frac{1}{2}$ (anal. 6) to $8\frac{1}{2}$ (anal. 7); in clinoptilolite this ratio ranges from about $6\frac{1}{2}$ (anal. 5) to nearly 11 (anal. 1). The compositions of these two minerals can only be expressed satisfactorily by formulas based on the unit cell content of 72 oxygen atoms.

Refractive indices for the analyzed heulandites and clinoptilolites, where available, are listed in the key to Fig. 2. These data indicate that refractive indices and birefringence decrease with increase in Si, both in heulandite and in clinoptilolite; substitution of Na and K for Ca results in a decrease of refractive index. Clinoptilolites have lower refractive indices than heulandites; from the available data no heulandite has a beta index lower than 1.488, and no clinoptilolite a beta index higher than 1.485. This provides a ready means for distinguishing the two minerals.

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CLINOPTILOLITE REDEFINED

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ABSTRACT

Since its introduction into the literature in 1923, clinoptilolite has been a subject of confusion within the zeolite group of minerals. A critical review is made of papers dealing with the original description of clinoptilolite, its occurrence in the groundmass of altered pyroclastics and of the arguments favoring its identity with "silica-rich" heulandite.

New experimental data on clinoptilolite from Hector, California, and on other samples from Tertiary sediments assist in the redefinition of this zeolite. Although the powder pattern of clinoptilolite is almost identical with that of heulandite, high temperature *x*-ray diffraction studies show that at about 230° C., heulandite transforms into "heulandite B," and at 350° C. becomes amorphous. Clinoptilolite, however, remains stable to about 700° C. without reaction at lower temperatures.

The data of chemical, optical and differential thermal analyses are also employed to redefine clinoptilolite as the high-silica member of the heulandite structural group usually containing monovalent rather than divalent cations, and to retain the name as a valid mineral species on equal status with heulandite.

The enrichment of silica in the clinoptilolite lattice as compared to heulandite is apparently correlatable with its increased thermal stability, and is probably a direct consequence of its origin from altered volcanic glass.

INTRODUCTION

The recent work of Ames, Sand and Goldich (1958) on the Hector, California bentonite deposit has disclosed the presence of a zeolite mineral in beds of altered pyroclastic material, closely associated with the montmorillonite, hectorite. This zeolite was identified by them as clinoptilolite, a mineral first named by Schaller in 1923. The exact distinction between clinoptilolite and heulandite, however, has been the subject of discussion for many years.

Hey and Bannister (1934) concluded from similarities in *x*-ray diffraction patterns that minerals previously reported as clinoptilolite were simply "silica-rich heulandites" and that there was little to justify use of the former name.

If the mineral first described by Pirsson in 1890 from the Hoodoo Mountains, Wyoming, and subsequently named clinoptilolite by Schaller (1933) were a unique occurrence, there would be little justification for a detailed redefinition of the species at this time. However, other occurrences of "heulandite-like" or "clinoptilolite-like" zeolites have been reported from the groundmass of altered pyroclastic beds and their lacustrine equivalents, not to mention the extensive heulandite-tuff horizons of South Island, New Zealand (Coombs, 1953). (See also Bramlette and Posnjak, 1933; Kerr, 1951; Kerr and Cameron, 1934; Regnier, 1958;

Deffeyes, 1958; Weeks, 1958; Weber, 1957, and Van Houten, 1959.) The relatively widespread distribution of heulandite-clinoptilolite zeolites would seem to necessitate a proper redefinition of such species and the properties by which one may readily distinguish them from each other.

The present study attempts to redefine the material clinoptilolite by first reviewing the published results of others and secondly by presenting new experimental data determined on heulandite and clinoptilolite samples. These data clearly indicate that clinoptilolite is a valid mineral species closely related to, but clearly distinct from, heulandite.

Previous Work

In 1890 the second occurrence of "mordenite" was reported by L. V. Pirsson from the Hoodoo Mountains of northwestern Wyoming. This zeolite was found in the amygdules of a weathered basalt and, despite its platy habit, was identified with the mordenite from Nova Scotia described by How (1864), primarily on the basis of its chemical analysis. Pirsson's analysis of optically homogeneous material (see Table 1) yields a base:alumina:silica:water mole ratio of 1:1:10:6.6 which is close to the 1:1:9:6 reported by How and the 1:1:10:7 ratio reported for ptilolite (Cross and Eakins, 1886). It occurs as small, platy crystals (1×1×4 mm.) unlike the habit of either Nova Scotia mordenite or the fibrous ptilolite for Colorado (Cross and Eakins, 1886). Crystallographically this zeolite was reported by Pirsson to be identical with heulandite. Its optical properties, however, are not completely consistent with those of mordenite (Schaller, 1932).

In reviewing Pirsson's description, Schaller (1932) concluded from new optical determinations, its platy habit and monoclinic crystallography, that the Hoodoo Mountain zeolite should be classified as neither mordenite or ptilolite, and instead, proposed the new name "clinoptilolite" for this species, alluding to its compositional similarity with ptilolite and its rather large extinction angle.

The widespread distribution of materials answering the above descriptions was first noted by Bramlette and Posjnak (1933) who pointed out that clinoptilolite is a common alteration product in pyroclastics where alteration has not proceeded as far as the formation of montmorillonite. Samples of Tertiary bentonite from San Luis Obispo County, California, Dome, Arizona and Pedro, Wyoming, were examined and in each instance the small crystals and almost isotropic relic shards from the washed residues could be identified optically and crystallographically with the clinoptilolite of Schaller. An analysis of the Dome material is close to that of clinoptilolite if allowance is made for about 5% clay impurity. A pure sample was not available, however, and the presence of quartz, feldspar, montmorillonite and unaltered volcanic glass is likely. For the first time, *x*-ray diffraction patterns of clinoptilolite were taken and although no *d* spacings were reported, the patterns were described by Bramlette and Posjnak as distinct from that of mordenite, but practically indistinguishable from that of heulandite.

The *x*-ray similarity between clinoptilolite and heulandite was further expanded by Hey and Bannister (1934) who themselves took rotation photographs around the *a* axis of a single crystal of clinoptilolite from the type locality, No. 5268, in the Brush Collection at Yale University. The results were "completely identical" with the *a* axis rotation photograph of heulandite. They explained the wide differences in optical properties between clinoptilolite and heulandite as due to slight difference in chemical composition and concluded that these zeolites must be members of the same isomorphous series (*sic*) and the name clinoptilolite is unnecessary.

Since the work of Bramlette and Posjnak (1933), zeolites resembling the clinoptilolite of Schaller have been described from numerous localities as alteration products of volcanic ash and pyroclastic beds. Kerr (1931) described rhyolitic glass from Ventura, California showing alteration to a weakly birefringent, platy zeolite whose indices of refraction matched closely those of heulandite. In a bentonite from Tehachapi, California, Kerr and Cameron (1936) noted a clear, colorless, platy zeolite in amounts up to ten per cent, also with low birefrigence and a mean index of refraction around 1.475. *X*-ray diffraction patterns of this material gave lines which could be identified with those of heulandite.

Also of significance is the heulandite reported by Fenner (1936) as groundmass cement in drill cores from Yellowstone, and by Steiner (1953) at the Hotsprings of Wairakei, New Zealand. Gilbert and McAndrews (1948) described an authigenic zeolite resembling either heulandite or clinoptilolite from a marine arkose in Santa Cruz County, California. They report that this material is unaffected by boiling 20 per cent HCl and has optical properties more closely approximating those of clinoptilolite than the literature values for heulandite. In addition they reported a significant difference in the effect of heating on the optical axis of their authigenic zeolite compared with normal heulandite.

Coombs (1954) has studied in detail the thick sequence of altered volcanic tuff near Southland, New Zealand of Triassic age and reports the presence of considerable amounts of zeolite minerals including a heulandite-like species. His chemical analysis of slightly impure material gives rise to a mole ratio of 1:1:6.2:5. This mineral occures in fibers or lamellae replacing volcanic glass shards. Coombs concludes that this mineral is "essentially a normal heulandite and not the silica-rich variety, clinoptilolite . . . which has been frequently reported in tuffs."

On the basis of chemical and optical data Ames, Sand and Goldich (1958) concluded that the tabular zeolite associated with hectorite at Hector, California, was identical with the clinoptilolite of Schaller. The "silica-rich heulandite" argument of Hey and Bannister (1934) was not considered by them even though the *x*-ray pattern of the Hector material matches well that of heulandite. Although the present author has noted the presence of up to 10 per cent fine-grained quartz and feldspar in the clinoptilolite samples kindly supplied by Drs. Ames and Sand from this locality, the above authors reported a mole ratio of 1:1:10:6 for this zeolite, as calculated from their chemical analyses. The mean index refraction of this material is 1.480, compared with 1.500 for heulandite.

In addition to the several dozen pyroclastic beds of the Western states sampled by the author, unpublished data kindly supplied by Drs. F. B. Van Houton, K. Deffeyes, J. Regnier and G. C. Kennedy, have led to the realization that clinoptilolite is not an uncommon alteration product of volcanic pyroclastics, and that a clear distinction between clinoptilolite and heulandite is imperative at this time.

EXPERIMENTAL RESULTS

During the course of a broad mineralogical study of natural and synthetic zeolites, a number of samples of heulandite and clinoptilolite have been examined. It should be especially noted that most of the samples referred to as clinoptilolite are actually clinoptilolite-rich rocks. Pure fractions of clinoptilolite are extremely difficult to separate and consequently much of the work on this mineral has been carried out on unbeneficiated samples, containing up to 25% quartz, feldspar and montmorillonite impurities. The samples from Hector, California, Fish Creek Mountains, Nevada and Patagonia are all at least 90% pure; the last sample appears to be 99% clinoptilolite. *X*-ray diffraction studies, optical examinations and thermal treatments, are, of course, little affected by small amounts of impurities. In addition, many significant trends may be ascertained from chemical analyses of slightly impure material even though exact empirical formulas may be calculated only from analyses of pure minerals.

X-Ray Diffraction and Optical Data

A sample labeled as Pirsson's type mordenite from Hoodoo Mountains, Wyoming, was borrowed from the Brush Mineral Collection of Yale University, No. 5268. The prominent phase of this specimen occurs as pink, radiating needles and showed the characteristic *x*-ray diffraction pattern of well-crystallized mordenite. The friable white matrix consisted of quartz and feldspar. Evidently, this specimen is not the same material examined by Hey and Bannister (1934) (*vide supra*). If no additional Hoodoo Mountain type material can be located, it is indeed difficult to interpret data previously determined on Pirsson's original material.

Drs. L. B. Sand and Lloyd Ames kindly supplied several samples of the material described by Ames, Sand and Goldich (1958) as clinoptilolite from Hector, California. In thin section the rock consists of about 20% cavities or voids (plainly visible in hand specimens) about 1–2 mm. in diameter, 70–75% weakly birefringent platy crystals less than 2 microns in size and about 10–15% quartz, feldspar, and montmorillonite impurities (see Fig. 1). The major component occurs as tiny, shredded plates or laths, replacing the glass shards, relicts of which are abundant in the section. In many places the clinoptilolite can be seen projecting out into cavities from the edges. The crystalline impurities are about .1–.5 mm. in size.

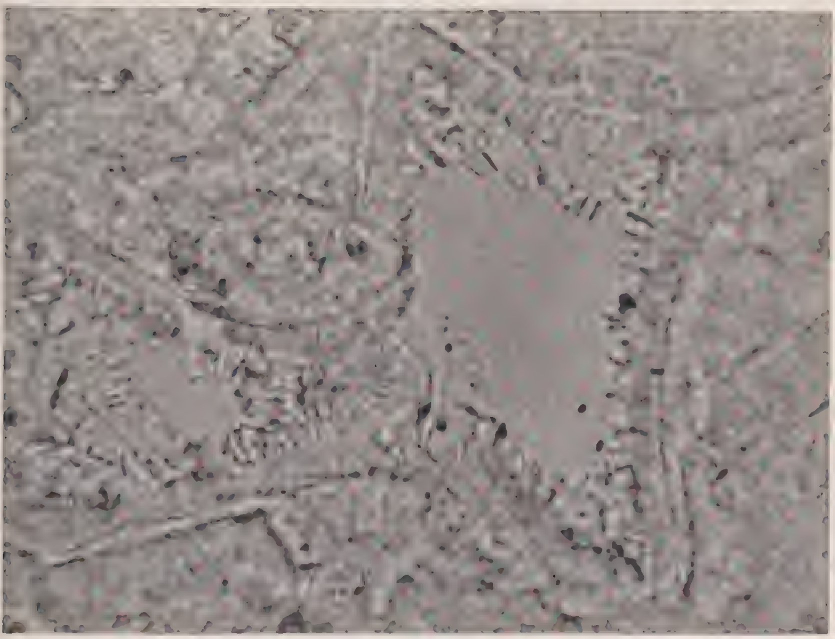

FIG. 1. Photomicrograph of altered volcanic tuff, Hector, California. ×700.

The x-ray diffractometer trace (Table 1, Fig. 2) is similar to that reported by Ames, Sand and Goldich (1958), both being very similar to heulandite. No feldspar peaks were observed and only a broad, diffuse peak could be measured at low angles, to indicate montmorillonite. The

TABLE 1. LATTICE SPACING FOR CLINOPTILOLITE AND HEULANDITE

Hector, California Clinoptilolite		Prospect Park, New Jersey Heulandite	
d	I/I_0	d	I/I_0
9.00	10	8.90	10
7.94	4	7.94	2
6.77	3	6.80	1
6.64	2	6.63	1
5.91	1	5.92	1
—	—	5.58	1
5.24	3	5.24	1
5.11	1	5.09	1
—	—	4.89	1
4.69	2	4.69	2
4.48	2	4.45	2
4.34	2	4.36	1
3.96	10	3.97	2
3.90	8	3.89	3
3.83	1	3.83	1
3.73	1	3.71	1
3.55	2	3.56	1
3.46	2	3.47	1
3.42	6	3.40	2
3.12	3	3.12	1
3.07	2	3.07	1
3.04	2	3.03	1
2.97	5	2.97	4
2.87	1	—	—
2.82	3	—	—
2.80	1	2.80	1
2.73	1	2.72	1
2.72	1	—	—
2.68	1	2.67	1
2.44	1	2.48	1
2.42	1	2.43	1
2.38	1	2.35	1
2.29	1	2.28	2

intense peak at 3.34 Å is coincident with the major quartz line. High temperature *x*-ray diffraction data given below prove conclusively that this is a quartz peak. Several fractions of the powdered sample were prepared by means of simple water sedimentations. Aside from an increase in the intensity of the montmorillonite peak in the lightest fraction, no other changes were observed.

In Fig. 2 are shown the *x*-ray diffractometer traces of heulandite from

Prospect Park, New Jersey, and clinoptilolite from Hector, California. These tracings are typical of many other samples examined by the author and the corresponding d spacings and intensities are listed in Table 1. The x-ray diffraction tracings of 7 typical heulandite and 8 clinoptilolite specimens are shown schematically in Fig. 3. The peaks of clinoptilolite are consistently much stronger than those of heulandite, and somewhat broader. Many of the weak peaks present in clinoptilolite tracings are absent in those of heulandites. The (020) reflection of heulandite at 9.9°2θ is always far more intense than the remaining lines of the pattern, while this same reflection in x-ray tracings of clinoptilolite is, in many samples, exceeded in intensity by the (004) peak at about 22.3°2θ. The many differences between diffractometer tracings of clinoptilolite and heulandite have proved quite adequate for the identification and differentiation between these two similar zeolites. From the spacing differences listed in Table 1, the unit cell of clinoptilolite appears to be slightly larger in the b dimension than heulandite (using the structural scheme of Ventriglia, 1955, for heulandite).

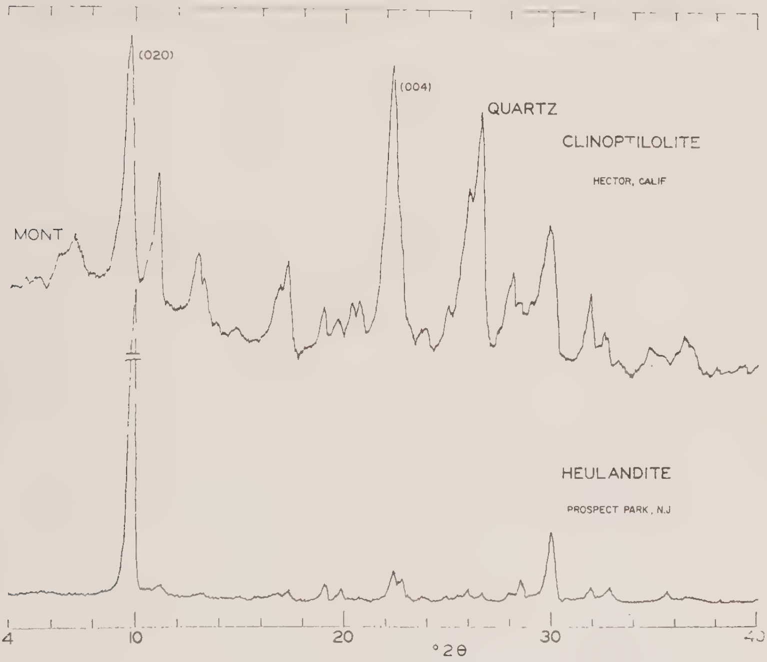

FIG. 2. X-ray diffraction tracings of heulandite and clinoptilolite.

The x-ray diffraction pattern published by Coombs (1958) of a cli-

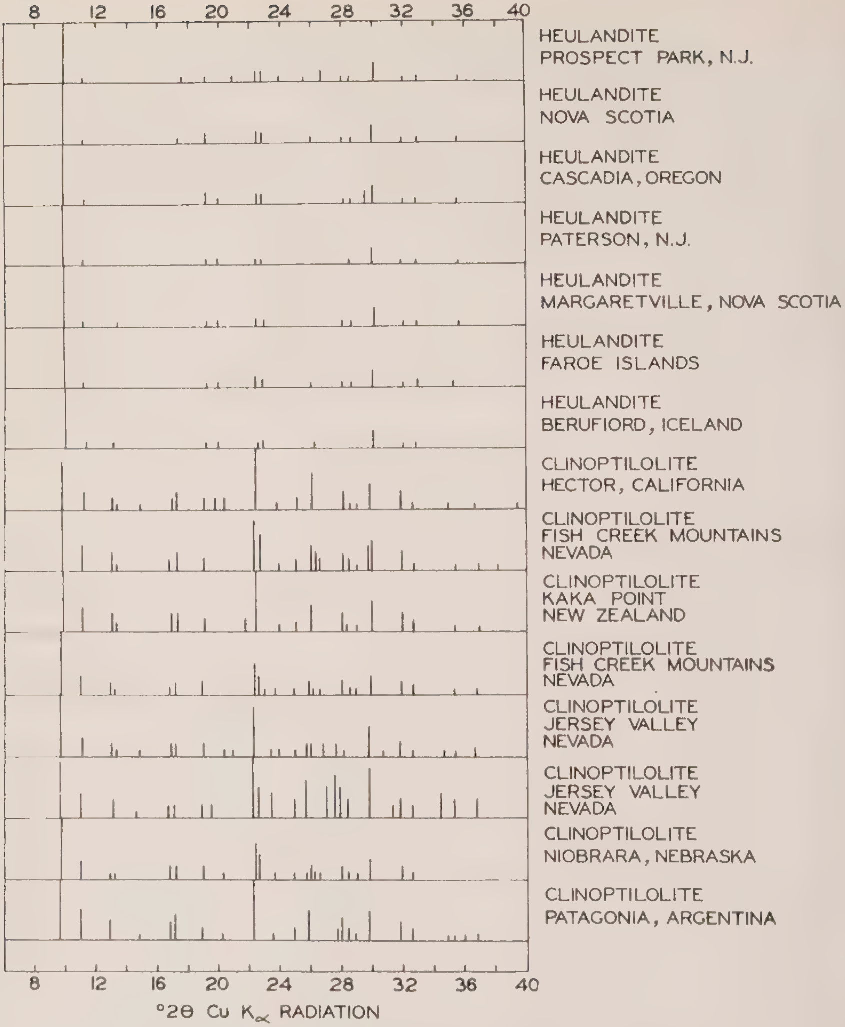

FIG. 3. Typical *x*-ray diffractometer patterns of heulandite and clinoptilolite (schematic).

noptilolite sample from Seaham, New South Wales also shows slightly greater *d* spacing unit cell and the typical clinoptilolite peak intensities, compared with heulandite.

Film patterns of heulandites and clinoptilolites are very similar and only with precise measurements can spacing differences between the two minerals be noted. The line intensities are also very similar, indicating that the packing of the sample for a spectrometer trace has resulted in a preferred orientation of heulandite grains. Most clinoptilolite grains are normally less than 1 micron in diameter and thus little orientation takes place when the sample holder is packed.

Thermal Data

Perhaps the strongest data supporting clinoptilolite as a valid mineral species, distinct from heulandite, are those determined by high temperature *x*-ray diffraction techniques, differential thermal analyses and thermal gravimetry. A heulandite from Prospect Park, New Jersey, and a clinoptilolite from Hector, California, were studied by the first of these methods in the following manner. The samples were powdered and packed into a platinum holder, which was then placed vertically in the *x*-ray beam of a Norelco 90° Diffractometer. A platinum-wound resistance furnace was placed around the sample holder and the temperature raised at intervals to 1200° C. The temperature was measured by chromel-alumel thermocouples, changed at frequent intervals, and could be controlled at ±5° C. for several hours and at ±10° C. overnight, at temperatures below 900° C. The results of the thermal *x*-ray study readily distinguish between heulandite and clinoptilolite.

No transitions or reactions were observed in clinoptilolite up to about 750° C. At this temperature the structure began to collapse after heating for 4 hours. Heating at higher temperatures resulted in the complete disappearance of the *x*-ray diffraction pattern and the formation of an amorphous product, resembling a glass. The prolonged stability of the peak at 3.34 Å, however, long after the clinoptilolite pattern had disappeared, indicates that this peak is the major quartz peak, an initial contaminant of the sample. Many quartz-free samples of clinoptilolite have been found whose *x*-ray tracings do not show this peak.

Heulandite, on the other hand, is far less stable to thermal treatment and undergoes a reaction at about 230±10° C. Above this temperature the phase present has been identified with the "heulandite B" of Slawson (1925) and Milligan and Weiser (1937) and Koizumi (1953).

In Fig. 4 are the typical differential thermal analysis patterns of heulandite and clinoptilolite. The patterns of a dozen other samples were all quite similar. Although the temperatures of the reactions are slightly greater than those determined by high temperature *x*-ray diffraction, they represent the same phenomena described above. Aside from the broad endothermic peak, clinoptilolite exhibits no further thermal reaction up to 1000° C. Heulandite, however, shows an additional very sharp endotherm at approximately 300° C. which undoubtedly corresponds to the sluggish heulandite-"heulandite B" reaction, shown to take place at 230° C. by the high temperature *x*-ray diffraction study.

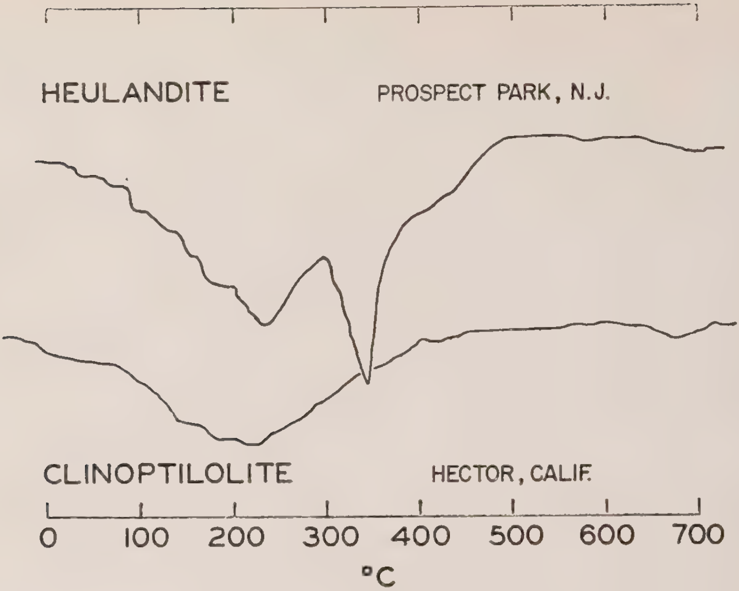

Fig. 4. DTA patterns of heulandite and clinoptilolite.

Thermal gravimetric analyses of heulandite at a heating rate of 40° per hour show a sharp steepening of the weight loss curve also in the region of 250° C. No break in the weight loss curve was noted for clinoptilolite.

Other authors have indicated thermal reactions for heulandite close to 120° C., but no evidence of such phenomena could be found in this study (see Milligan and Weiser, 1937).

If the differences in thermal stability between heulandite and clinoptilolite are due solely to the nature of the exchange cation in each species (*vide infra*) one might expect a calcium-exchanged clinoptilolite to exhibit the low temperature reactions and low thermal stability of heulandite. Conversely, a sodium-exchanged heulandite should be more thermally stable than the calcium variety. Such is not the case. Clinoptilolite from Hector, California, was almost completely exchanged to the calcium form by repeated slurrying with a large excess of a 1 Normal calcium chloride solution. A chemical analysis of the washed product gave 6.4 wt-% CaO, 0.2 wt-% Na_2O. If it is assumed that no exchange of the potassium and magnesium occurred, the clinoptilolite product is greater than 90% calcium exchanged. The *d* spacings and intensities of the peaks are virtually identical with those of the original clinoptilolite. A sodium-rich form of heulandite could not be prepared by the above methods even when boiling sodium chloride solutions were used.

Differential thermal analysis of the calcium exchanged clinoptilolite resulted in a pattern insignificantly different from that of the original sodium-potassium form, indicating that Ca-clinoptilolite does not transform to a "B" phase at low temperatures and is thermally stable well above the decomposition temperature of Ca-heulandite. This material was heated overnight at 450° C. and examined by *x*-ray diffraction methods. Only slight, if any, loss of structure was noted from the diffraction tracing.

At 550° C., after 24 hours, most of the structure of the Ca-clinoptilolite was destroyed although the main *x*-ray diffraction peaks were still discernible.

These data indicate that, although the calcium form of clinoptilolite is somewhat less stable than the sodium-potassium form, Ca-clinoptilolite does not resemble heulandite in its *x*-ray diffraction pattern nor in its low thermal stability. The presence of additional "structural" silica in the clinoptilolite lattice, compared with heulandite, appears to be more responsible for its increased stability than the nature of the exchange cation.

Chemical Composition

According to Pirsson's original analysis of the Hoodoo Mountain material, clinoptilolite has the molecular formula 1:1:9:6.8, base:alumina:silica:water, considerably more silica than most heulandites. In addition the dominant cation in heulandites is calcium, while this analysis listed almost equal parts of lime, soda and potash.

Due to both the fine grain character of almost every other clinoptilolite sample collected from sediments of pyroclastic origin, and the intimate association of the zeolite with admixed montmorillonite, opal and volcanic glass fragments, it has been almost impossible to separate a pure fraction of the zeolite for chemical analysis. It has been equally difficult to determine the purity of such a mixture after a particular separation attempt, since both optical and *x*-ray diffraction methods are of little use. Little confidence, therefore, can be placed on empirical formulas derived from the literature analyses of clinoptilolite, since the purity of the samples is uncertain. The analysis of "pure" clinoptilolite sample has recently been reported by Ames, Sand and Goldich, although the *x*-ray diffraction pattern of this material indicates the presence of several per cent quartz. In thin sections of Hector clinoptilolite, about 10 per cent quartz can be distinguished.

Mason (personal communication) reports chemical data for a clinoptilolite sample from Patagonia which lead to the empirical formula, $(.04\ MgO\ .07\ CaO\ .64\ Na_2O\ .25\ K_2O)Al_2O_3\ 10.8\ SiO_2\ 6.5\ H_2O$. By *x*-ray diffraction and optical examinations, this material appears quite pure, probably containing less than 1% impurities. Electron microscopy, however, reveals the presence of trace amounts of a foreign phase.

The meager chemical data in the literature indicate that clinoptilolite has a silica/alumina ratio of about 8.5 to 10.5 and contains predominantly monovalent over divalent cations.

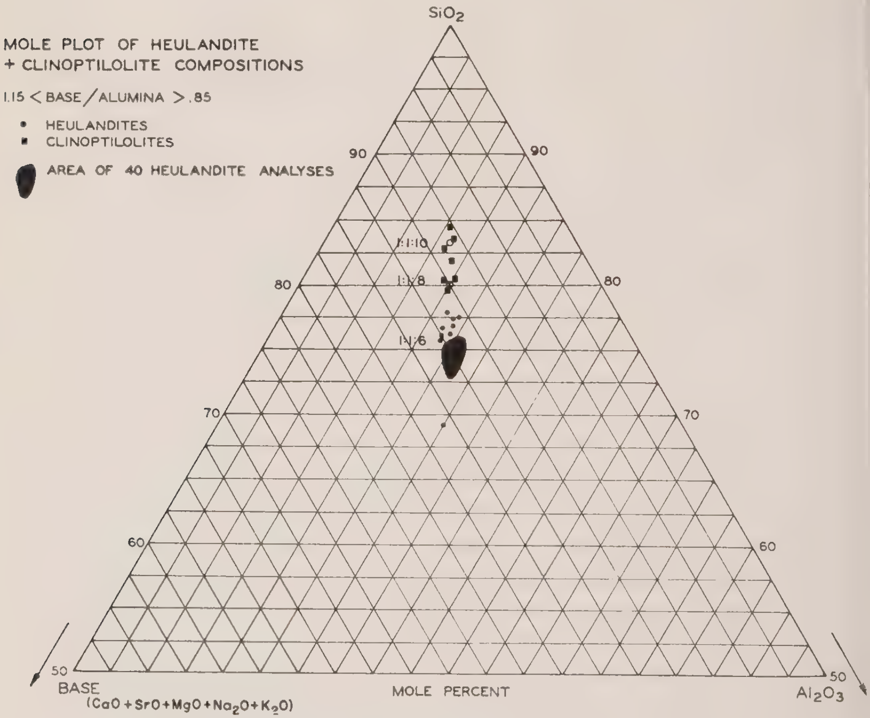

FIG. 5. Mole compositions of heulandites and clinoptilolites plotted on the ternary diagram base-alumina-silica.

The mole compositions of nearly 50 selected heulandite analyses are plotted in Fig. 5. The distribution of silica/alumina ratios for these literature analyses is shown in Fig. 6. From both figures it can be seen that the SiO_2/Al_2O_3 ratio of heulandites varies from 5.4 to 6.7, with only a few analyses greater than 6.7 (the accuracy of which are unknown).

Although not as conclusive as the differences in silica content, heulandites and clinoptilolites may also be differentiated on their water content. The number of moles of water per mole of alumina in heulandites varies between 5 and 6 while in clinoptilolite this ratio is usually between 6 and

7 (see Fig. 6). It is also interesting to note the difference in the so-called exchange cations of these two similar zeolites. In Fig. 7 are plotted the exchange cation compositions of heulandites and the few clinoptilolites available. It can be seen that heulandites are overwhelmingly calcium-rich while clinoptilolites contain major proportions of sodium and potassium. (Many heulandite analyses list considerable SrO, which has been added to the CaO for simpler calculations.)

At first glance it may seem plausible to distinguish these two minerals on the basis of the differences in their exchange cations. A distinction based solely on these grounds is, however, quite inadequate and can only be secondary to the much more significant difference in the silica content. The cation content of a zeolite is determined primarily by the cation environment at the time of crystallization and secondarily by the composition of the ground waters passing over it since that time. It is unlikely that ground waters could change the cation composition of heulandite although clinoptilolite might well be influenced by them. It is not unlikely that in the future a calcium form of clinoptilolite will be uncovered in nature, possibly in an environment much like the New Zealand hot springs locality described by Steiner (1953) and the tuffs described by Coombs (1954).

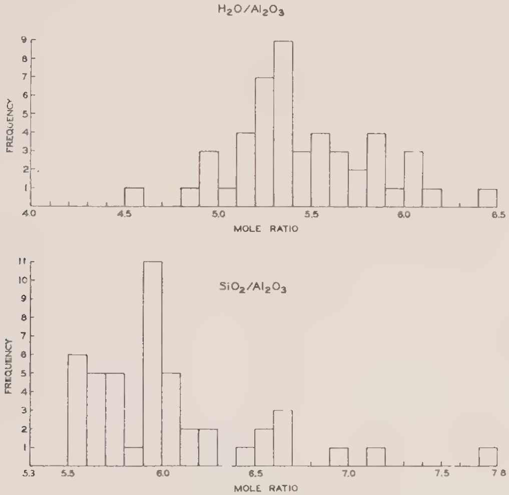

FIG. 6. Frequency distribution of mole ratios for heulandites. (1.15 < base/alumina > 0.85)

There is no precedent for naming two mineral species, which differ only on their exchangeable cation content. Individual names are not given to the many varieties of montmorillonite simply because the exchangeable cations are different from one specimen to another. Montmorillonoid minerals have been classified on the basis of the difference in nature of the so-called "structural" cations, that is, the ions of the tetrahedral and octahedral layers. It seems reasonable then to differentiate heulandite and clinoptilolite by the differences in their "structural" ions –the significant variation in the silica and alumina contents of their unit cells.

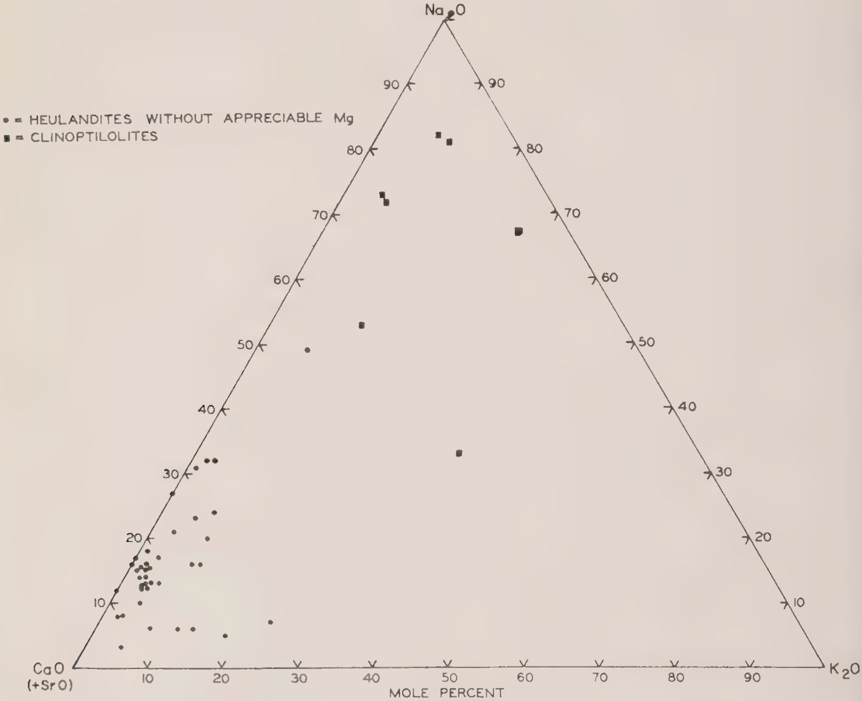

FIG. 7. Mole plot of base distribution in heulandites and clinoptilolites.

Despite the isomorphous relationship between the structures of heulandite and clinoptilolite there is little evidence to suggest the existence of complete solid solution between these two minerals. Chemical analyses and other properties indicate two distinct zeolite species.

To use the chemical analyses of minerals from the literature in support of a particular hypothesis is quite risky and only overwhelming evidence in favor of a proposed theory can be considered acceptable. If only a few scattered analyses of heulandites were to be presented, the existence of a continuous solid solution between the mole ratios 1:1:5 and a 1:1:10, base:alumina:silica, would appear plausible. If, however, many more analyses are calculated and plotted, as in Figs. 5 and 6, such a hypothesis cannot be accepted on the evidence available. Despite the so-called ideal unit cell ratio of 1:1:7, as determined by Wyart (1933), the analyses of natural minerals favor a range of 5.5 to 6.5 for the silica/alumina ratios of heulandites.

Clinoptilolite may, therefore, be defined chemically as the high silica member of the heulandite structural group usually containing more monovalent rather than divalent cations.

Such differences in the silica/alumina ratio of zeolites of the same structural type are, of course, quite significant and can be correlated directly with acid and thermal stabilities. Recent unpublished studies in this laboratory suggest that the silica content of certain zeolites is a direct function of the chemical environment from which these zeolites crystallized. Analcite, phillipsite, and erionite from igneous rocks all contain lesser amounts of silica in their structures than do their "sedimentary" equivalents. This may be due to the nature of their formation from volcanic glass particles by the action of ground waters. The availability of large amounts of silica in the glass combined with the presence of soda and potash in both the glass and the attacking waters may account for the subsequent high-silica, high-soda content of the zeolite.

TABLE 2. HYDROTHERMAL STABILITY OF CLINOPTILOLITE

Temperature ° C.	Pressure psi	Duration Days	Products
		*Clinoptilolite, Hector, California**	
300	20,000	8	Clinoptilolite+some Mordenite+Quartz
355	15,000	15	Mordenite+some Montmorillonite+Quartz
395	20,000	17	Montmorillonite+Albite+Quartz
		Clinoptilolite, Patagonia	
300	20,000	8	Clinoptilolite
355	15,000	15	Mordenite
445	20,000	7	Albite+Cristobalite+?

* Quartz and a small amount of montmorillonite are present in starting material.

*Hydrothermal Data**

According to Buckner (1958) clinoptilolite transforms to mordenite at 300° C. and 20,000 psi water vapor pressure. This reaction has been repeated by the author on three samples of clinoptilolite, the results of which are listed in Table 2. All samples were reacted in sealed gold tubes containing a drop of distilled water. The samples did not react in identical manners under the same pressure-temperature conditions, possibly reflecting small differences in composition of the clinoptilolites. At 300° C., the Hector material begins to transform into mordenite and analcite while the Patagonia specimen remains stable after 8 days. At 355° C. both are almost completely converted to mordenite. Since the starting material contained an abundance of soda, sodium mordenite was produced, together with small amounts of silica. An envelope run at 300° C. of the Hector material resulted in the formation of analcite, quartz and some montmorillonite, indicating a possible leaching of silica from the starting material, which does not occur in the sealed tubes. Above 400° C. hydrothermal treatment of clinoptilolite resulted in the formation of alkali feldspars and silica. These data suggest that the upper limit of "stability" of clinoptilolite under hydrothermal conditions is probably about 300° C. or less.

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* Hydrothermal studies were carried out using standard methods and equipment previously described by Roy and Roy (1955) and Roy, Roy and Osborn (1953).

Dr. M. Koizumi (personal communication) has found that heulandite decomposes to wairakite only, above 320° C. and under 15,000 psi water pressure.

Discussion and Conclusions

From the cursory descriptions and investigations on clinoptilolite available in the literature, it was not wholly inaccurate to label clinoptilolite as simply a silica-rich heulandite. Upon closer examination, however, there seems to be little doubt that clinoptilolite is a zeolite mineral closely related to, but distinct from, heulandite in composition, properties, stability and genesis. The occurrences of this zeolite in the groundmass of altered pyroclastic sediments will no doubt lead to speculations upon its genesis from the glass shards of volcanic ash and tuff. Because of the fact that monomineralic samples of clinoptilolite are almost impossible to obtain and that this zeolite usually occurs as laths about one micron or less in size, several of the standard mineralogical constants such as specific gravity or optical properties, cannot easily be determined with any degree of certainty.

The optical properties of zeolites in general are difficult to obtain since small variations in hydration, cation content or silica/alumina ratio all seem to affect the optical crystallography of the mineral. From the published data and the new samples studied in this investigation, clinoptilolite has a mean index of refraction of about 1.480 compared to 1.500 of most heulandites, slightly larger values for the optical angle and very low birefringence. Otherwise, the optics and crystallography of both minerals appear to be quite similar.

The similarity in optical properties and external crystallography suggest that the structures of clinoptilolite and heulandite are also closely related. The unit cell constants of clinoptilolite are only slightly larger than those of heulandite. Spectrometer traces of the two minerals are easily differentiated by the presence of many intense diffraction peaks for clinoptilolite and the somewhat larger *d* spacings of this mineral as compared with heulandite.

The most decisive criteria for distinguishing one phase from another are their reactions to thermal treatment. The low thermal stability and the reaction at 250° C. to a "B" modification clearly differentiates heulandite from clinoptilolite. The latter zeolite is thermally stable to well over 700° C. and goes through no low temperature reactions or transitions. That the higher thermal stability of clinoptilolite is not caused by the nature of the exchange cation is borne out by the stability of Ca-exchange clinoptilolite at 450° C., compared with the low temperature decomposition of Ca-heulandite. These conclusions are corroborated by both high temperature *x*-ray diffraction and differential thermal analysis data as well as by the results of thermal gravimetry. The high thermal stability of clinoptilolite more closely approximates those of the "rigid three-dimensional framework" zeolites such as analcite, mordenite, chabazite, and the Linde synthetic 4A and 13X zeolites all of which are thermally stable to at least 700° C. Heulandite, on the other hand, breaks down at relatively low temperatures, similar to the low thermal stability of "layered" and "fibrous" zeolites, natrolite, thomsonite, stilbite, and phillipsite. It is possible that the additional silica present in clinoptilolite has provided the "layered" structure of heulandite with additional structural linkages, thereby strengthening the framework.

Although analyses of heulandites are readily obtained, the difficulty in separating a pure clinoptilolite fraction from its host rock prohibits accurate chemical analyses. By correcting several analyses of clinoptilolite for the impurities present, silica/alumina ratios of about 9–10 can be calculated. The silica/alumina ratios of the heulandites studied in this investigation in addition to those compiled from the literature, group predominantly around 6.0. Essentially no heulandites or clinoptilolites have been found whose chemical compositions or physical properties approach what might be called a continuous solid solution series between the two end members, even if differences in their cation content are neglected.

Although the structures of these two zeolites are isomorphous, solid solution between heulandite, at a composition of 1:1:6:6 and clinoptilolite at 1:1:9–10:6, does not seem to be complete. It seems only proper, therefore, to classify clinoptilolite as a separate mineral species. On the other hand, it is likely that the silica content of clinoptilolite samples does vary between about 8.5 and 10.5.

Orginally the name clinoptilolite was coined to indicate its chemical similarity with ptilolite and its inclined extinction. Although chemical and physical properties are obviously much closer to heulandite than either ptilolite or mordenite, the name clinoptilolite already has been used on numerous occasions in the literature to represent the zeolite found in the groundmass of altered pyroclastics, and whose properties approximated those of heulandite. For this reason, it seems advisable to retain the name clinoptilolite despite the ambiguity of its origin.

In summary, clinoptilolite can be defined as a mineral of the zeolite group having a molecular composition close to $(Na_2O)_{.70}(CaO)_{.10}(K_2O)_{.15}(MgO)_{.05}\cdot Al_2O_3\cdot 8.5\text{–}10.5\ SiO_2\cdot 6\text{–}7\ H_2O$ and a structure similar to heulandite but which can be clearly distinguished from heulandite by optical, x-ray, thermal and chemical means.

Coupled with x-ray diffractometer traces, thermal treatment affords the best means by which heulandite and clinoptilolite may be clearly differentiated. X-ray film patterns are not suitable for differentiating between the two species. The DTA pattern of heulandite contains a sharp endotherm in the vicinity of 300° C. in addition to the zeolitic dehydration endotherm at lower temperatures. Clinoptilolite shows only the dehydration endotherm at low temperatures. If DTA equipment is not available, one need only place the sample in question in an oven at 450° C. overnight and examine the product by x-ray diffraction. Under these conditions the pattern of heulandite will have disappeared, and that of clinoptilolite will be essentially unaffected.

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CORRELATION OF SOME PHYSICAL PROPERTIES AND CHEMICAL COMPOSITION OF SOLID SOLUTION

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Abstract

The tacit assumption for the linear correlation of the density and the chemical composition of a solid solution is twofold: one is an ideal solid solution and the other identical or nearly identical molar volumes of the components. In an ideal solid solution, the difference in densities obtained by the non-linear and linear treatments are discussed. These are applied to the spessartite-almandite, almandite-pyrope, forsterite-fayalite and wurtzite-greenockite systems. The garnet systems can be treated practically by the linear correlation, but the olivine and the wurtzite systems must be treated with caution. Coefficients of thermal expansion and compressibilities of the ideal solid solution can be discussed in the same manner. But when the solid solution is ideal, the linear correlation of molar heat capacities and chemical composition is possible. The linear correlation of refractive index and chemical composition of an isotropic solid solution can be derived from the Gladstone-Dale equation, but it is required that the system must be ideal and the molar volumes of the components are equal or nearly equal. If the concept of the volume fraction is introduced, density, coefficient of thermal expansion, compressibility and refractive index can be correlated linearly with the volume fraction in an ideal system.

I. Density

Density has been correlated linearly for the determination of the chemical composition of a solid solution. Bloss (1952), however, found out that this is not actually true in an ideal binary solid solution. This can be proved more rigorously in a multicomponent system by a simple thermodynamical discussion. The difference between the densities obtained by the non-linear and linear treatments in the ideal system is of considerable interest to us. Since we usually use the linear correlation for the determination of the composition, we want to know the maximum density difference from non-linear correlation, so the accuracy of the density measurements for the linear correlation can be stated. See Fig. 1

Zen (1956*a*) discussed the validity of Vegard's law in an ideal cubic solid solution and concluded that unless molar volumes of the components are equal or nearly equal, the law does not hold. A similar situation arises in the correlation of density and chemical composition of a solid solution. The importance of the molar volumes of the components in the correlation has been pointed out by Bloss. He suggested that the negative and positive deviations of the non-linear treatment from that of the linear depend entirely upon the ratio of the molar volumes of the components in an ideal binary solid solution.

In the m component solid solution, the mass, M, is given by

$$M = \sum_{i}^{m} n_i \rho_i \bar{V}_i \qquad (1)$$

where n_i, ρ_i and $\bar{V}_i$ are the number of moles, the density and the molar volumes of the ith component of the solid solution, respectively. The volume of the solid solution, V, is

$$V = \sum_i^m n_i \left(\frac{\partial V}{\partial n_i}\right)_{nk} = \sum_i^m n_i \bar{\bar{V}}_i \tag{2}$$

where $\bar{\bar{V}}_i$ is the partial molar volume of the ith component, and k denotes all the components except the ith. The volume can be also expressed by

$$V = \bar{V} \sum_i^m n_i \tag{3}$$

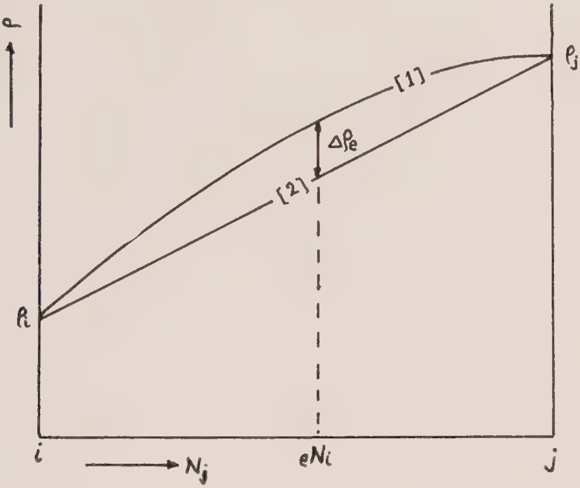

FIG. 1. Correlation of density and chemical composition of an ideal binary solid solution. The curve [1] is the non-linear correlation and [2] linear approximation. For other symbols, see the text. The example is given for the case of $\Delta\rho_e > 0$, which is termed the positive deviation from linearity. When the error of the density measurement is larger than $\Delta\rho_e$, the [2] correlation can be used approximately instead of [1]. If the density measurement is designed to be more accurate and the error is smaller than $\Delta\rho_e$, the correlation should be more properly made by the non-linear curve [1]. It is noticed that $eNi \approx 0.5$.

where $\bar{V}$ is the molar volume of the solid solution. The division of (1) by (2) gives the density of the solid solution, that is,

$$\rho = \frac{1}{\bar{V}} \sum_i^m N_i \rho_i \bar{V}_i . \tag{4}$$

Also

$$\rho = \frac{1}{\sum N_i \bar{\bar{V}}_i} \sum N_i \rho_i \bar{V}_i \tag{4'}$$

since

$$\frac{n_i}{\sum_i^m n_i} = N_i$$

where N_i is the mole fraction of the ith component. This procedure of deriving (4) is essentially the same as that of Bloss. But (4) can be applied for both cases, ideal or non-ideal solid solution systems.

In an ideal solid solution we can set

$$\bar{V}_i = \bar{\bar{V}}_i. \tag{5}$$

Therefore, we have

$$\rho = \frac{1}{\sum N_i \bar{V}_i} \sum N_i \rho_i \bar{V}_i. \tag{6}$$

If the densities of all the components happen to be same, from (6)

$$\rho = \rho_i.$$

Of course, in this case, the density measurements fail to determine the chemical composition of the solid solution. Instead, if the molar volumes of the components are identical, from (6)

$$\rho = \sum_i^m N_i \rho_i \tag{7}$$

since

$$\sum_i^m N_i = 1.$$

Equation (7) indicates the linear relation between the density and chemical composition. Thus the tacit assumptions for the linear correlation are: an ideal solid solution and identical molar volumes of the components. However, this is not new and only confirms Bloss' finding.

We now consider the ideal system in which the densities of the components are not identical, nor the molar volumes. In an ideal solid solution, the difference between the densities given by the non-linear and linear treatments is as follows.

$$\Delta\rho = \frac{\sum N_i \rho_i \bar{V}_i}{\sum N_i \bar{V}_i} - \sum N_i \rho_i. \tag{8}$$

The function, $\Delta\rho$, has at least one extremum (maximum or minimum). Though no detailed mathematical proof is given here, this is quite clear since $\Delta\rho = 0$ at the pure end members. The N_i at which $\Delta\rho$ has the extremum can be solved by transformation of mole fraction in (8) into number of moles, differentiating both sides of the equation with respect to n_i and setting

$$\left(\frac{\partial(\Delta\rho)}{\partial n_i}\right)_{nk} = 0.$$

The reason for this transformation is obvious from the independent property of number of moles. Arranging the results with the reverse transformation of n_i into N_i, we have

$$\bar{V}_i \frac{\rho_i \sum N_i\bar{V}_i - \sum N_i\rho_i\bar{V}_i}{\rho_i - \sum N_i\rho_i} = (\sum N_i\bar{V}_i)^2. \tag{9}$$

However, it must be noted that in the binary system only one equation is necessary, but two for the ternary system and so forth. This can be easily understood by the property of the mole fraction.

For simplicity, only the solution for the binary system is given here. Suppose the two components are i and j, respectively, and the required mole fraction is eN_i. Then, from (9)

$$(eN_i)^2(\bar{V}_i - \bar{V}_j) + 2eN_i\bar{V}_j - \bar{V}_j = 0 \tag{10}$$

The solution of the above quadratic equation is given by

$$eN_i = \frac{\sqrt{\bar{V}_i\bar{V}_j} - \bar{V}_j}{\bar{V}_i - \bar{V}_j} \tag{11}$$

It is quite surprising to see the eN_i at which $\Delta\rho$ has the extremum is not a function of densities, but of the molar volumes of the components. The negative sign in front of the square root of (11) has been dropped, since $0 \leq N_i \leq 1$.

We know

$$\frac{\bar{V}_i + \bar{V}_j}{2} > \sqrt{\bar{V}_i\bar{V}_j}$$

in (11). Thus if

$$\frac{\bar{V}_i + \bar{V}_j}{2}$$

is substituted into (11) in the place of $\sqrt{\bar{V}_i\bar{V}_j}$, $eN_i = \frac{1}{2}$ is obtained. Therefore, eN_i approaches $\frac{1}{2}$, as $\bar{V}_i$ approaches $\bar{V}_j$, or vice versa, because

$$\frac{\bar{V}_i + \bar{V}_j}{2} \approx \sqrt{\bar{V}_i\bar{V}_j},$$

when $\bar{V}_i \approx \bar{V}_j$. This is why Bloss found in some of his studies that the mole fraction at which the deviation is extremum is about 0.5. (Fig. 1)

If eN_i determined by (11) is substituted into (8), this will give us the extremum deviation of the non-linear treatment from the linear $\Delta\rho_e$, that is,

$$\Delta\rho_e = \frac{\sqrt{\overline{V}_i} - \sqrt{\overline{V}_j}}{\sqrt{\overline{V}_i} + \sqrt{\overline{V}_j}} (\rho_i - \rho_j) \tag{12}$$

According to (12), the function, $\Delta\rho_e$, may be positive or negative depending on the sign of $(\sqrt{\overline{V}_i} - \sqrt{\overline{V}_j})$ and $(\rho_i - \rho_j)$.

The following examples are given under the assumption of ideality except in the case of the forsterite-fayalite system. This is somewhat arbitrary, since we do not have experimental data to support this. However, this is the simplest way for the purpose of the present discussion.

Example 1. Spessartite-Almandite System

According to Skinner (1956), the densities are 4.190 and 4.318 for spessartite and almandite, respectively. Also the molar volumes are 118.15 cc./mole for spessartite and 115.28 for almandite. From equation (11), the mole fraction of spessartite, eN_i, is found to be 0.49_8 which is quite close to 0.5. The deviation of density $\Delta\rho_e$ is -0.000_6. Thus the deviation is almost insignificant compared to the accuracy of the present density measurements and the linear correlation can be justified.

Example 2. Almandite-Pyrope System

Skinner gives, for pyrope, the density, 5.582 and the molar volume, 113.29 cc./mole. The desired mole fraction of almandite eN_i is found to be 0.49_7 and the extremum deviation $\Delta\rho_e$ is -0.003_2. By the same reason as the former example, the linear correlation is possible, if the error in the density measurements is greater than 0.003_2g/cc.

Example 3. Forsterite-Fayalite System

Yoder and Sahama (1957) concluded from the heat of solution and additivity of the molar volumes that this system belongs to an ideal system within the accuracy of the measurements. They give the densities, 3.222 for synthetic forsterite and 4.392 for synthetic fayalite. The molar volumes are also given, 43.7cc./mole for forsterite and 46.4 for fayalite. The desired mole fraction for fayalite eN_i is 0.49_2 and the deviation $\Delta\rho_e$ is $+0.017_6$. The deviation is rather high. This can be explained if we look at the equation (12). That is, when the molar volumes of the components are small and the density difference is large, the deviation becomes larger. As shown already, the maximum deviation of the density is about 0.5%. Thus caution will be necessary in using the linear correlation.

Example 4. Wurtzite-Greenockite System

Hurlbut (1957) noticed that the density data of the intermediate compositions in this system fall below the straight line in the linear correlation, though the difference is not much. The data for the present calculation are taken from Hurlbut. Wurtzite has the density, 4.1, $a_0 = 3.811$ Å and $c_0 = 6.234$ Å, and greenockite, the density, 4.9, $a_0 = 4.142$ Å and $c_0 = 6.724$ Å.

The number of formula weights in a unit cell is two. Using Avogadro number $N = 6.023 \times 10^{23}$, the molar volumes calculated are 23.6 cc./mole for wurtzite and 30.1 for greenockite. The desired mole fraction of wurtzite eN_i is 0.47_7 and the maximum difference between the non-linear and the linear treatment, $\Delta\rho_e$ is $+0.048_7$ in density, which is about 1.1% deviation from linearity. The conclusion starting from the ideality of the solid solution gives the positive deviation but the experimental data contradict this, that is, the data by Hurlbut give the negative deviation. If the negative deviation is true, the $\sum N_i \overline{V}_i < \sum N_i \overline{\overline{V}}_i$ from (8). Thus the volume increase can be expected by the mixing according to (4). However, the calculation of the molar volumes of the intermediate and end members of the synthetic wurtzite-greenockite system from his data indicates a volume decrease by mixing. If the density of greenockite is chosen to be 4.8, which is calculated from his x-ray data for synthetic greenockite, instead of the measured value of 4.9, his density data show the positive deviation as the present theory predicts. However, a further study is necessary in this respect.

II. Coefficient of Thermal Expansion, Compressibility and Heat Capacity

In an ideal m component solid solution, we have

$$\bar{V} = \sum_{i}^{m} N_i \bar{V}_i \tag{13}$$

Differentiating the molar volumes with respect to temperature at constant pressure or with respect to pressure at constant temperature, the following equations can be obtained.

$$\begin{aligned} \left(\frac{\partial \bar{V}}{\partial T}\right)_p &= \sum N_i \left(\frac{\partial \bar{V}_i}{\partial T}\right)_p \\ \left(\frac{\partial \bar{V}}{\partial p}\right)_T &= \sum N_i \left(\frac{\partial \bar{V}_i}{\partial p}\right)_T \end{aligned} \tag{14}$$

However,

$$\begin{aligned} \alpha &= \frac{1}{\bar{V}}\left(\frac{\partial \bar{V}}{\partial T}\right)_p, \qquad & \alpha_i &= \frac{1}{\bar{V}_i}\left(\frac{\partial \bar{V}_i}{\partial T}\right)_p \\ \beta &= \frac{1}{\bar{V}}\left(\frac{\partial \bar{V}}{\partial p}\right)_T, \qquad & \beta_i &= -\frac{1}{\bar{V}_i}\left(\frac{\partial \bar{V}_i}{\partial p}\right)_T \end{aligned} \tag{15}$$

Then the suitable substitution of (15) into (14) gives

$$\alpha = \frac{1}{\sum N_i \bar{V}_z} \sum N_i \alpha_i \bar{V}_i \tag{16}$$

$$\beta = \frac{1}{\sum N_i \bar{V}_i} \sum N_i \beta_i \bar{V}_i \tag{17}$$

where α and α_i are the coefficients of thermal expansion of the solid solution and the ith component, and β and β_i are the compressibilities. Here again, if the molar volumes of the components are equal or nearly equal, (16) and (17) can be reduced into

$$\alpha = \sum_i^m N_i \alpha_i \tag{18}$$

$$\beta = \sum_i^m N_i \beta_i \tag{19}$$

Therefore, the analogous equations such as (8) to (12) can be constructed in the correlation of coefficients of thermal expansion and compressibilities with chemical composition. Equation (11) shows that the mole fraction of the ith component at which the deviation of density from the linear is maximum, is a function of molar volumes of the components only, thus the mole fraction is common in the cases of correlation of density, coefficient of thermal expansion and compressibility with the chemical composition of the solid solution.

The molar heat content, H, of a solid solution can be expressed by

$$H = \sum N_i \left(\frac{\partial H}{\partial n_i}\right)_{nk} = \sum N_i \bar{H}_i \tag{20}$$

where $\bar{H}_i$ is the partial molar heat content of the ith component. Differentiating both sides of (20) with respect to temperature at constant pressure, we have

$$\left(\frac{\partial H}{\partial T}\right)_p = \sum N_i \left(\frac{\partial^2 H}{\partial_T \partial n_i}\right)_{nk}$$

Exchanging the order of differentiation in the right hand side of the above equation and from

$$\left(\frac{\partial H}{\partial T}\right)_p = C_p,$$

which is the molar heat capacity.

$$C_p = \sum N_i \left(\frac{\partial}{\partial n_i} C_p\right) = \sum N_i \bar{C}_{pi} \tag{21}$$

where $\bar{C}_{p_i}$ is the partial molar heat capacity of the ith component. In an ideal system

$$C_{pi} = \bar{C}_{pi} \tag{22}$$

Thus we can have

$$C_p = \sum N_i C_{pi} \tag{23}$$

Therefore, the correlation of the molar heat capacity with the chemical composition of a solid solution is linear as far as the solid solution is ideal. Apparently few experimental data exist for the discussion of Part II.

III. Refractive Index

In general practice, the index of refraction of an isotropic solid solution is linearly represented as follows.

$$\gamma = \sum_i^m N_i \gamma_i \tag{24}$$

where γ and γ_i are the refractive indices of m component solid solution and its ith component, and N_i is the mole fraction of the ith. Equation (24) has been also generalized for anisotropic solid solutions. According to Barth (1930), the equation is analogous to the second theoretical equation by Mallard which is the same as that obtained by Duffet empirically. The tacit assumption in his practice will be studied from the property of refractivity. The total refractivity, R, is expressed by

$$R = \frac{\gamma^2 - 1}{\gamma^2 + 2} V \qquad \text{(Lorentz-Lorenz)} \tag{25}$$

or

$$R = (\gamma - 1)V \qquad \text{(Gladstone-Dale)} \tag{26}$$

The calculation of the index of refraction from the density data has been attempted by several authors (e.g., Larsen (1921), Jaffe (1956) and Allen (1956)), though Barth stated that the relation between the two is not simple. However, the empirical equation by Gladstone-Dale rather than the theoretical one by Lorentz-Lorenz generally appears to be in the best agreement with the observed facts (Eitel (1954)).

We define the partial molar refractivity of the ith component of the solid solution as follows.

$$\bar{R}_i = \left(\frac{\partial R}{\partial n_i}\right)_{nk} \tag{27}$$

And generalization of the equation by Guggenheim (1957) gives

$$R = \sum_i^m n_i \bar{R}_i \tag{28}$$

The differentiation of both sides of Lorentz-Lorenz equation (25) with respect to n_i gives

$$\bar{R}_i = \frac{\gamma^2 - 1}{\gamma^2 + 2} \bar{\bar{V}}_i \tag{29}$$

From (25), (28) and (2), with transformation of n_i into N_i,

$$\sum N_i\bar{R}_i = \frac{\gamma^2 - 1}{\gamma^2 + 2}\sum N_i\bar{\bar{V}}_i \tag{30}$$

Call R_i the molar refractivity of the ith component and then the following equation is valid.

$$\sum N_i R_i = \sum N_i \frac{\gamma_i^2 - 1}{\gamma_i^2 + 2}\bar{V}_i \tag{31}$$

The division of (30) by (31) gives

$$\frac{\sum N_i\bar{R}_i}{\sum N_i R_i} = \frac{\dfrac{\gamma^2 - 1}{\gamma^2 + 2}\sum N_i\bar{\bar{V}}_i}{\sum N_i \dfrac{\gamma_i^2 - 1}{\gamma_i^2 + 2}\bar{V}_i} \tag{32}$$

If the deviation of ideality is not great in a solid solution, then

$$\frac{\sum N_i\bar{R}_i}{\sum N_i R_i} \approx 1 \tag{33}$$

Thus

$$\frac{\gamma^2 - 1}{\gamma^2 + 2} \approx \frac{1}{\sum N_i\bar{\bar{V}}_i}\sum N_i \frac{\gamma_i^2 - 1}{\gamma_i^2 + 2}\bar{V}_i \tag{34}$$

Equation (34) may be expanded as follows.

$$\begin{aligned}\left(1 - \frac{2}{\gamma^2} + \frac{4}{\gamma^4} - \frac{8}{\gamma^6} + \frac{16}{\gamma^8}\cdots\right) &\approx \frac{1}{\sum N_i\bar{\bar{V}}_i} \\ &= \sum N_i\left(1 - \frac{2}{\gamma_i^2} + \frac{4}{\gamma_i^4} - \frac{8}{\gamma_i^6} + \frac{16}{\gamma_i^8}\cdots\right)\bar{V}_i\end{aligned} \tag{35}$$

Then we can have

$$\left.\begin{aligned}\frac{1}{\gamma^2} &\approx \frac{1}{\sum N_i\bar{\bar{V}}_i}\sum N_i \frac{1}{\gamma_i^2}\bar{V}_i \\ \frac{1}{\gamma^4} &\approx \frac{1}{\sum N_i\bar{\bar{V}}_i}\sum N_i \frac{1}{\gamma_i^4}\bar{V}_i \\ &\vdots\end{aligned}\right\} \tag{36}$$

These are obtained by equating the terms of both sides of (36), which have the same power of r and r_i. This method indicates also $\sum N_i\bar{\bar{V}}_i \approx \sum N_i\bar{V}_i$ from the first term. This can be understood from (33). If this is an ideal solid solution and the molar volumes of the components are equal or nearly equal, equation (36) becomes

$$\frac{1}{\gamma^l} = \sum N_i \frac{1}{\gamma_i^l}, \qquad l = 2q, \qquad q = 1, 2, 3, \cdots \tag{37}$$

However, when $q=1$ in the equation above, we have

$$\frac{1}{\gamma^2} = \sum N_i \frac{1}{\gamma_i^2} \tag{38}$$

which is analogous to the first equation given by Mallard theoretically in 1884 for the binary cubic mixed crystals. Barth (1930) believes that Mallard's theory depends on the analogy of the phenomena produced by the superposition of thin crystalline plates. It is proved that Mallard's equation can be derived from Lorentz-Lorenz equation. And the equations for $q>1$ should be tested by experiments.

From Gladstone-Dale equation,

$$\bar{R}_i = (\gamma - 1)\bar{\bar{V}}_i \tag{39}$$

Under the same assumption and with the same procedures as before, we have

$$\gamma \approx \frac{\sum N_i \gamma_i \bar{V}_i}{\sum N_i \bar{\bar{V}}_i} \tag{40}$$

When the solid solution is ideal, (40) becomes

$$\gamma = \frac{\sum N_i \gamma_i \bar{V}_i}{\sum N_i \bar{V}_i} \tag{41}$$

In addition, if the molar volumes of the components are equal or nearly equal, (41) can be reduced into (24). The linear correlation can be derived from Gladstone-Dale equation under the condition of the ideal solid solution and identical or nearly identical molar volumes of the components. The similarity of equation (41) to those of density, coefficient of thermal expansion, and compressibility equations is obvious. Therefore, it is suggested that the analogous equation such as (8) to (12) may be constructed in the correlation of the refractive index and the chemical composition of the solid solution. But equation (41) should be tested by experiments. The simplest test may be the measurements of the refractive indices of an ideal binary solution.

SUMMARY AND CONCLUSION

The simple linear correlation of the density and the refractive index with the chemical composition of a solid solution which is found in standard textbooks in mineralogy was analyzed in Part I and Part III. In Part I, the conservation of mass and additivity of partial molar volumes are the basis of the discussion. In Part II, additivity of molar volumes (in an ideal system) and that of partial molar heat content are utilized. In Part III, the discussion was conducted from additivity of partial molar refractivity, which is not a thermodynamical quantity and also from that of partial molar volumes.

In an ideal system, equations (6), (16), (17) and (41) indicate that these physical properties of the components are weighted to their proportion of volumes and then give the average properties of the solid solution. As already shown, these equations can be summarized as follows.

$$\frac{\sum N_i\rho_i\bar{V}_i}{\rho} = \frac{\sum N_i\alpha_i\bar{V}_i}{\alpha} = \frac{\sum N_i\beta_i\bar{V}_i}{\beta} = \frac{\sum N_i\gamma_i\bar{V}_i}{\gamma} = \sum N_i\bar{V}_i \tag{42}$$

We define the volume fraction, v_i, in the following manner.

$$v_i = \frac{N_i\bar{V}_i}{\sum N_i\bar{V}_i} \tag{43}$$

Thus

$$\sum_i^m v_i = 1 \tag{44}$$

In terms of the volume fraction, (42) can be expressed by

$$\begin{aligned} \rho &= \sum \rho_i v_i \\ \alpha &= \sum \alpha_i v_i \\ \beta &= \sum \beta_i v_i \\ \gamma &= \sum \gamma_i v_i \end{aligned} \tag{45}$$

In other words, these physical properties can be correlated linearly with the volume fraction of the components. When the molar volumes of the components happen to be identical, the volume fraction is identical with the mole fraction. This can be shown from (43). As shown by (44) and (45), measurements of one physical property is sufficient to determine the volume fraction in an ideal binary solid solution, two for an ideal ternary system and so on. One might prefer the use of "degree of freedom" in this kind of discussion. But, according to the present discussion, the determination of the lattice constant is only involved in equation (44). Thus this does not introduce a new physical property to determine the volume fraction, that is to say, the determination of the lattice constant does not reduce the degree of freedom of the system to determine the volume fraction. On the contrary, the lattice constant determination can be used for the conversion of the volume fraction into the mole fraction. For simplicity, suppose a cubic ideal solid solution, then

$$\bar{V} = \frac{Na^3}{Z}, \qquad \bar{V}_i = \frac{Na_i^3}{Z} \tag{46}$$

where a and a_i are the lattice constants of the solid solution and its ith component, N is Avogadro number and Z is number of the formula in a unit cell. No structure change is assumed in this solid solution. Equation (46) is substituted into (13), thus we have

$$a^3 = \sum N_i a_i^3 \tag{47}$$

Therefore, the volume fraction can be expressed by

$$v_i = N_i \left(\frac{a_i}{a}\right)^3 \tag{48}$$

And the conversion of volume fraction into mole fraction can be done by the following equation.

$$N_i = \left(\frac{a}{a_i}\right)^3 v_i \tag{48'}$$

Regression equations relating chemical compositions and physical properties have been prepared for dolomite by Zen (1956b) and for anthophyllite by Hey (1956). Hey emphasized certain inherent limitations for their use, that is, a regression equation can only be utilized for one particular purpose. An equation or set of equations derived to predict the physical properties at the given chemical composition, can not properly be used to predict the chemical compositions at the given physical properties. This restriction in regression equations is lucidly illustrated by Moroney (1956). If it is desired to predict the chemical composition, given an adequate number of physical data, a set of regression equations must be calculated for this purpose (e.g., Zen (1957)), as Hey asserts.

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LAYER CHARGE RELATIONS IN THE DIOCTAHEDRAL AND TRIOCTAHEDRAL MICAS*

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ABSTRACT

In theoretical formulas for dioctahedral and trioctahedral micas, the entire composite-layer charge originates in the tetrahedral layers owing to substitution of one-fourth of the Si by Al; the octahedral layer is neutral. But formulas calculated for published analyses of a great number of dioctahedral and trioctahedral micas show that these ideal charge relations are altered by the proxying of other cations for the octahedral cation theoretically present. Cations carrying a lower positive charge than the cation ideally presumed to occupy the octahedral sites, as Mg or Li for Al in dioctahedral micas, or Li for Mg or Fe^{+2} in trioctahedral micas, do not proxy for that cation in sufficient number to compensate for the difference in charge; consequently, not all the negative octahedral charges are neutralized, and the layer is left with a negative charge which necessitates an equivalent decrease in the tetrahedral negative charge by increase in Si and decrease in Al. In such micas, part of the total composite layer charge originates in the octahedral layer, part in the tetrahedral layer, and the total charge is the sum of two negative charges.

The additional charges carried by cations having a higher charge than the octahedral cations for which they proxy, as Al or Fe^{+3} for Mg in trioctahedral micas, may be accommodated in two ways: (a) by a positive charge on the octahedral layer, which is compensated for by an equivalent increase in the negative tetrahedral charge by greater substitution of Al for Si, or (b) by nonoccupancy of octahedral sites, the negative charges associated with these sites being then available to neutralize the additional positive charges carried by the proxying cations. Formulas of natural trioctahedral micas show that in most of them accommodation has been made in both ways to varying degrees; in very few has accommodation been made entirely in one way. In most trioctahedral micas, therefore, the total charge is made up of a positive octahedral charge and a negative tetrahedral charge greater than 1.00 by an equivalent amount.

INTRODUCTION

In the ideal formulas for the prototypes of the dioctahedral and trioctahedral trisilicic micas, muscovite and phlogopite, the octahedral layer is neutral, and the entire inherent charge on the composite layer originates in the tetrahedral layer owing to the proxying of trivalent aluminum for quadrivalent Si. The octahedral layer is neutral. This octahedral-tetrahedral charge relation can be shown by a notation above the respective groups in the structural formulas:

$$\overset{.00}{\mathrm{Al}_{2.00}}\overset{-1.00}{(\mathrm{Si}_{3.00}\mathrm{Al}_{1.00})}\mathrm{O}_{10}(\mathrm{OH})_2]^{-1.00}\ \overset{+1.00}{\mathrm{K}}_{1.00}$$

$$[\overset{.00}{\mathrm{Mg}_{3.00}}\overset{-1.00}{(\mathrm{Si}_{3.00}\mathrm{Al}_{1.00})}\mathrm{O}_{10}(\mathrm{OH})_2]^{-1.00}\ \overset{+1.00}{\mathrm{K}}_{1.00}$$

However, very few, if any, natural muscovites and phlogopites have the exact composition specified in these ideal formulas. Almost all muscovites contain a little magnesium, and almost all phlogopites contain a little octahedral aluminum, Al(VI). Both muscovites and phlogopites may contain some iron, trivalent, bivalent, or both. Some also have titanium, or manganese, and more rarely, vanadium and chromium. These cations occupy octahedral sites and proxy for the ion specified in the ideal formulas.

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If the proxying cation has the same charge as the cation for which it proxies, there is no change in the number of positive charges carried by the octahedral cations, and the neutrality of the octahedral layer is not affected. But if the proxying cation has a different charge than the cation for which it proxies, the octahedral layer acquires either a positive or a negative charge, depending on whether the proxying cation carries a higher or a lower charge than the proxied cation. The effect of such substitutions, based on calculated formulas of published analyses, was studied in detail for dioctahedral micas (Foster, 1956), trioctahedral micas (Foster, in press), and for lithium micas (Foster, in press). The present paper represents a correlation of the results of these studies with respect to layer charge relations.

The Effect of Substitutions in Dioctahedral Micas

Although two octahedral sites are ideally occupied by trivalent cations (as two Al in the ideal formula of muscovite) formulas calculated from published analyses of dioctahedral micas show that some of the octahedral sites are occupied by quadrivalent cations, such as Ti, and divalent cations, such as Fe^{+2}, Mg and Mn, or even univalent cations, such as Li.

Replacement by quadrivalent cations

Quadrivalent cations proxying for trivalent cations, ion for ion, increase the number of positive octahedral charges and necessitate layer charge adjustments. Thus substitution of 0.25 Ti^{+4} for 0.25 Al^{+3}(VI)* in muscovite:

$$[\overset{+.25}{(Al_{1.75}Ti_{.25})}\overset{-1.25}{(Si_{2.75}Al_{1.25})}O_{10}(OH)_2]^{-1.00}\overset{+1.00}{K}_{1.00}$$

induces a positive charge of 0.25 on the octahedral layer and necessitates an equivalent increase in the negative charge on the tetrahedral layer by decrease in Si content and increase in Al content, in order to preserve a composite layer charge of 1.00. However, no analysis of a dioctahedral mica has come to hand in which Ti alone or so much Ti replaces Al. In most calculated formulas of dioctahedral micas the amount of Ti is much lower than 0.25, and usually divalent cations more than compensate for the effect of Ti.

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* Al(VI) indicates octahedral Al; Al(IV) indicates tetrahedral Al.

Replacement by bivalent cations

Most commonly the cations proxying for Al in the dioctahedral micas in addition to Fe^{+3}, are the bivalent cations, Fe^{+2}, Mg, and Mn^{+2}. A bivalent cation proxying for a trivalent octahedral cation, ion for ion, decreases the number of positive octahedral charges, and some of the negative charges associated with the octahedral layer are left unneutralized. Thus the layer has a negative charge, which necessitates adjustment in the tetrahedral layer. In order to preserve a composite-layer charge of 1.00, the negative charge on the tetrahedral layer must decrease by an amount equivalent to the negative charge on the octahedral layer, that is, there must be an increase in Si and a decrease in Al(IV), in accordance with the equation, $R^{+2}+Si=Al(VI)+Al(IV)$. For example, in the following formula,

$$\Big[\underbrace{\overset{-.30}{(Al_{1.70}\overset{+2}{Fe}_{.08}Mg_{.22})}}_{2.00}\overset{-.72}{(Si_{3.28}Al_{.72})}O_{10}(OH)_2\Big]^{-1.02}K^{+1.01}_{1.01},$$

0.30 R^{+2} cations can be considered as having replaced 0.30 Al(VI) cations and 0.28 Si cations as having replaced 0.28 Al(IV) cations, 0.30 $R^{+2}+0.28\ Si=0.30\ Al(VI)+0.28\ Al(IV)$. The total composite-layer charge is a sum of two negative charges. Because bivalent cation replacement in the octahedral layer is more general and usually greater than quadrivalent cation (Ti) replacement, most formulas calculated from analyses of dioctahedral micas are characterized by (*a*) negative octahedral charge, (*b*) a tetrahedral charge less than -1.00, (*c*) a Si occupancy greater than 3.00, and (*d*) Al(IV) occupancy less than 1.00.

If half the trivalent octahedral cations ideally occupying the octahedral layer in muscovite are replaced by R^{+2} cations, the octahedral layer would have a charge of -1.00, and the tetrahedral layer would be entirely occupied by Si cations, and neutral.

$$\Big[\overset{-1.00}{(\overset{+3}{R}_{1.00}\overset{+2}{R}_{1.00})}\overset{.00}{Si_{4.00}}O_{10}(OH)_2\Big]^{-1.00}K^{+1.00}_{1.00}.$$

Schaller (1950) postulated such trisilicic-tetrasilicic series to explain the composition of high-silica sericites, and selected leucophyllite as the tetrasilicic end member. However, from available analyses, leucophyllite is not a mica, but a hydrous mica, with an H_2O content of 6.30 per cent and a K_2O content of only 3.39 per cent; interlayer cations occupy only 0.46 sites and the composite layer charge is only (-0.43), not (-1.00). The only representative of a true tetrasilicic dioctahedral potassium mica found among published analyses is a celadonite from near Reno, Nevada (analysis 5, Table 1), whose analysis yields the formula,

$$\overset{-.98}{\underbrace{[(Al_{.07}Fe^{+3}_{.93}}_{1.00}\,\underbrace{Fe^{+2}_{.24}Mg_{.77})}_{1.01}}\overset{.00}{Si_{4.00}}O_{10}(OH)_2{}^{-.98}(K, Na)^{+.98}_{.98}$$

In this formula the octahedral R^{+3} cations occupy one site, the R^{+2} cations 1.01 sites, and Si occupies exactly 4.00 sites. The tetrahedral layers are neutral and the entire composite layer charge originates in the octahedral layer.

TABLE 1. ANALYSES OF DIOCTAHEDRAL MICA REPRESENTING STEPS IN THE TRISILICIC-TETRASILICIC SERIES

	1	2	3	4	5
SiO_2	46.77	49.16	52.58	49.53	55.61
Al_2O_3	34.75	30.81	23.56	5.84	.79
Fe_2O_3	.71	—	—	20.06	17.19
FeO	.77	1.43	5.76	5.95	4.02
MgO	.92	2.22	2.43	2.92	7.26
MnO	—		—	—	.09
CaO	.13	.15	.65	.56	.21
Na_2O	.47	.48	—	.46	.19
K_2O	10.61	10.90	9.52	9.31	10.03
TiO_2	.21	.04	—	—	—
H_2O^-	4.48	.15	5.94	4.91	.48
H_2O^+		4.73			4.40
F	.16	—	—	—	—
	100.11	100.07	100.44	99.54	100.27
0=F	.07				
Total	100.04				

1. Sultan Basin, Snohomish Co., Washington; Axelrod and Grimaldi (1949), p. 565. Total includes 0.13% BaO.
2. Amelia, Virginia; Glass (1935), p. 756, no. 5.
3. Wildschapback-Thal, Baden, Germany; Dana (1892), p. 618, no. 45.
4. Urals, Russia; Hendricks and Ross (1941), p. 692, no. 5.
5. Reno, Nevada, 23 mi. E.; Wells (1937), p. 102, O.

The entire range of R^{+2} replacement is represented among natural dioctahedral micas, as shown by the analyses and formulas in Tables 1 and 2. The change in the seat of the composite layer charge from the tetrahedral to the octahedral layers is illustrated by means of histograms in Fig. 1. Formulas calculated from many published analyses of dioctahedral micas indicate that, throughout the series, octahedral occupancy remains constant at about 2.00 sites (in the half-cell formula) and that replacement of R^{+3} by R^{+2} is ion for ion. In general, the trisilicic end of the series is characterized by Al as the predominant trivalent octahedral cation, whereas the tetrasilicic end of the series is characterized by Fe^{+3}.

Replacement by univalent ions

In attempting to explain muscovites having as much as 3.3 per cent of $\mathrm{Li_2O}$, Levinson (1953, p. 93) concluded that, because of similarity in atomic radii, Al = 1.43 Å, Li = 1.51 Å, substitution of Li for Al is possible but not necessary, as Li could occupy vacant octahedral positions. Li occupying vacant octahedral sites in muscovite would cause a positive charge on the octahedral layer, which would in turn have to be compensated for by an equivalent increase in the negative charge on the tetrahedral layer by decrease in silicon and increase in aluminum, and the number of octahedral sites occupied would be greater than 2.00 by an amount equal to the number of sites occupied by Li. However, formulas calculated for analyses of lithian muscovites exhibit quite the contrary layer-charge relations, being characterized by a negative octahedral charge and by a decrease in the negative tetrahedral charge equivalent to the negative octahedral charge, as illustrated in the following formulas,

TABLE 2. FORMULAS FOR DIOCTAHEDRAL MICAS REPRESENTING STEPS IN THE TRISILICIC-TETRACILICIC SERIES (CALCULATED FROM ANALYSES IN TABLE 1)

1. Muscovite $\overset{-.10}{\underbrace{[(\mathrm{Al}_{1.84}\overset{+3}{\mathrm{Fe}}_{.04}\overset{+2}{\mathrm{Fe}}_{.04}\mathrm{Mg}_{.09})}_{2.01}}\overset{-.89}{(\mathrm{Si}_{3.11}\mathrm{Al}_{.89})}\mathrm{O}_{10}(\mathrm{OH})_2]^{-.99}(\mathrm{K}_{.90},\ \mathrm{Na}_{.06},\ \mathrm{Ca}/2_{.01})^{+1.00}_{.98}$

2. Sericite $\overset{-.30}{\underbrace{[(\mathrm{Al}_{1.70}\overset{+2}{\mathrm{Fe}}_{.08}\mathrm{Mg}_{.22})}_{2.00}}\overset{-.72}{(\mathrm{Si}_{3.28}\mathrm{Al}_{.72})}\mathrm{O}_{10}(\mathrm{OH})_2]^{-1.02}(\mathrm{K}_{.93},\ \mathrm{Na}_{.06},\ \mathrm{Ca}/2_{.01})^{+1.02}_{1.00}$

3. $\overset{-.49}{\underbrace{[(\mathrm{Al}_{1.45}\overset{+2}{\mathrm{Fe}}_{.33}\mathrm{Mg}_{.25})}_{2.03}}\overset{-.43}{(\mathrm{Si}_{3.57}\mathrm{Al}_{.43})}\mathrm{O}_{10}(\mathrm{OH})_2]^{-.92}(\mathrm{K},\ \mathrm{Ca}/2)^{+.92}$

4. Glauconite $\overset{-.72}{\underbrace{[(\mathrm{Al}_{.18}\overset{+3}{\mathrm{Fe}}_{1.12}\overset{+2}{\mathrm{Fe}}_{.37}\mathrm{Mg}_{.32})}_{1.99}}\overset{-.33}{(\mathrm{Si}_{3.67}\mathrm{Al}_{.33})}\mathrm{O}(\mathrm{OH})_2]^{-1.05}(\mathrm{K}_{.88},\ \mathrm{Na}_{.07},\ \mathrm{Ca}/2_{.02})^{+1.03}_{.99}$

5. Celadonite $\overset{-.98}{\underbrace{[(\mathrm{Al}_{.07}\overset{+3}{\mathrm{Fe}}_{.93}\overset{+2}{\mathrm{Fe}}_{.24}\mathrm{Mg}_{.77})}_{2.01}}\overset{.00}{\mathrm{Si}_{4.00}}\mathrm{O}_{10}(\mathrm{OH})_2]^{-.98}(\mathrm{K}_{.92},\ \mathrm{Ba}_{.03},\ \mathrm{Ca}/2_{.02})^{+1.01}_{.99}$

1. $\overset{-.10}{\underbrace{[(\mathrm{Al}_{1.89}\overset{+3}{\mathrm{Fe}}_{.01}\mathrm{Li}_{.20})}_{2.10}}\overset{-.91}{(\mathrm{Si}_{3.09}\mathrm{Al}_{.91})}\mathrm{O}_{10}(\mathrm{OH})_2]^{-1.01}(\mathrm{K}_{.88},\ \mathrm{Na}_{.07},\ \mathrm{Rb}_{.05},\ \mathrm{Cs}_{.01})^{+1.01}_{1.01}$

2. $$\Big[\overset{-.13}{\underbrace{(\mathrm{Al}_{1.73}\overset{+2}{\mathrm{Fe}}_{.07}\mathrm{Mg}_{.01}\mathrm{Mn}_{.02}\mathrm{Li}_{.48})}_{2.31}}\overset{-.89}{(\mathrm{Si}_{3.11}\mathrm{Al}_{.89})}\mathrm{O}_{10}(\mathrm{OH})_2\Big]^{-1.02}(\mathrm{K}_{.86},\ \mathrm{Na}_{08},\ \mathrm{Rb}_{.06},\ \mathrm{Cs}_{.01})^{+1.01}_{1.01}$$

3. $$\Big[\overset{-.20}{\underbrace{(\mathrm{Al}_{1.58}\overset{+3}{\mathrm{Fe}}_{.01}\overset{+2}{\mathrm{Fe}}_{.02}\mathrm{Mg}_{.01}\mathrm{Mn}_{.12}\mathrm{Li}_{.73})}_{2.47}}\overset{-.84}{(\mathrm{Si}_{3.16}\mathrm{Al}_{.84})}\mathrm{O}_{10}(\mathrm{OH})_2\Big]^{-1.04}(\mathrm{K}_{.82},\ \mathrm{Na}_{.10},\ \mathrm{Rb}_{.08},\ \mathrm{Cs}_{.01})^{+1.01}_{1.01}$$

which were calculated from the analyses given in Table 3. Also significant in these formulas is the number of sites occupied by octahedral cations, which exceed 2.00 by an amount considerably less than the number of sites occupied by Li. These layer-charge relations and octahedral occupancies indicate that Li has not simply occupied vacant octahedral sites, but has replaced Al(VI) at a ratio of less than three Li for one Al(VI). If three Li had replaced one Al(VI), there would have been no change in the layer-charge relations but a somewhat greater increase in octahedral occupancy. Assuming that the "primary" muscovites from which the lithian muscovites whose formulas appear above were derived to have had an octahedral occupancy of 2.00, the Li/Al(VI) replacement ratios in these formulas are two Li for one Al(VI), 2.8 Li for one Al(VI) and 2.8 Li for one Al(VI), respectively. These ratios are characteristic of the replacement ratios found in other lithian muscovites studied.

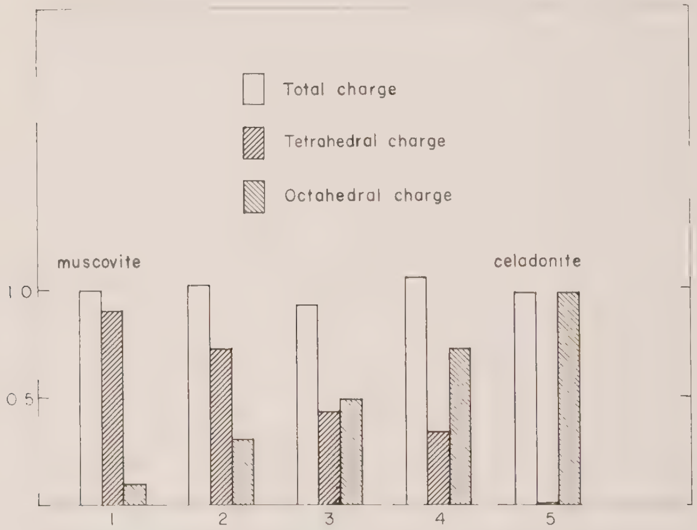

FIG. 1. Histograms of formulas of dioctahedral micas representing steps in the trisilicic-tetrasilicic series. (Numbers below histograms refer to formulas in Table 2.)

The micas whose formulas are given above were found by Levinson to have normal muscovite structures. From his study, Levinson concluded that the muscovite structure can accommodate up to about 3.3 per cent Li_2O. Micas having between 3.4 to 4.0 per cent Li_2O he found to have a combination of forms, 2-layer muscovite and 6-layer lepidolite, and those with more than 4.0 per cent Li_2O he found to have lepidolite structures. Correlation of these findings with calculated formulas of analyses of lithium-bearing micas, some the same ones as were studied by Levinson, showed that those having a combination of muscovite and lepidolite forms had octahedral occupancies of about 2.50 to 2.60, and an Li occupancy of about 0.90 to 1.05. Thus the muscovite structure persists to an octahedral occupancy of about 2.50 sites, halfway between the 2.00 sites of typical dioctahedral micas and the ideal three sites of trioctahedral micas.

TABLE 3. ANALYSES OF LITHIAN MUSCOVITES

	1	2	3
SiO_2	46.17	46.30	47.00
Al_2O_3	35.57	33.08	30.60
Fe_2O_3	.15	.00	.26
FeO	.08	1.20	.41
MgO	.00	.14	.13
MnO	.04	.28	2.04
CaO	.00	.00	Trace
Li_2O	.76	1.80	2.70
Na_2O	.56	.63	.77
K_2O	10.37	10.09	9.52
Rb_2O	1.1	1.37	1.93
Cs_2O	.3	.41	.18
H_2O^-	.12	.34	.25
H_2O^+	4.06	3.06	2.18
F	.76	2.06	4.09
	100.04	100.76	102.06
O=F	.32	.87	1.72
Total	99.72	99.89	100.34

1. Varuträsk, Sweden; Berggren (1941), p. 264, J.
2. Varuträsk, Sweden; Berggren (1940), p. 185, E.
3. Manitoba, Canada; Stevens (1938), p. 615, no. 1 (U. S. Nat. Museum no. 97635).

The composition of lepidolites can be interpreted in the same way as that of the lithian muscovites, with similar Li/Al(VI) replacement ratios and charge relations. However, they are not a continuation of that series, as they have a different structural form, being trioctahedral, not dioctahedral micas.

THE EFFECT OF SUBSTITUTIONS IN TRIOCTAHEDRAL MICAS

In the trioctahedral micas the most common substitutions are trivalent cations, Al and Fe^{+3}, or bivalent cations, Fe^{+2} or Mn, for bivalent Mg in the ideal phlogopite formula. Proxying bivalent cations, of course, have no effect on the layer-charge relations, but proxying trivalent cations tend to give a positive charge to the octahedral layer, which necessitates adjustments in the tetrahedral layer.

Replacement by trivalent cations

In Winchell and Winchell (1951), p. 373, the formulas for eastonite and siderophyllite (recast in the form used in this paper) assume that Al proxies for

$$[\overset{+.50}{(Al_{.5}Mg_{2.5})}\overset{-1.5}{(Si_{2.5}Al_{1.5})}O_{10}(OH)_2]^{-1.00}K^{+1.00}_{1.00}, \quad \text{and}$$

$$[\overset{+.50}{(Al_{.5}Fe^{+2}_{2.5})}\overset{-1.50}{(Si_{2.5}Al_{1.5})}O_{10}(OH)_2]^{-1.00}K^{+1.00}_{1.00},$$

for Mg or Fe^{+2} ion for ion. A substitution of 0.5 Al for 0.50 Mg or Fe^{+2} thus increases the number of positive octahedral charges by 0.5. This amount of positive charge on the octahedral layer necessitates an equivalent increase in the negative charge on the tetrahedral layers, so that the amount of Si present must decrease by 0.5 and the amount of Al must increase by the same amount. Octahedral occupancy remains constant at 3.00. However, Holzner (1936) noticed that in formulas calculated from analyses of biotites octahedral occupancy is usually between 2.89 and 2.49. To explain this low octahedral occupancy he postulated that biotite is intermediate between phlogopite and muscovite in composition, and that the crystal structure is built up of the two types, with most biotites approximating the ratio of two of phlogopite to one of muscovite, which yields a formula for biotite of

$$\underbrace{[\overset{.00}{(Al_{.67}Mg_{2.00})}}_{2.67}\overset{-1.00}{(Si_{3.00}Al_{1.00})}O_{10}(OH)_2]^{-1.00}K^{+1.00}_{1.00}.$$

In this formula the tetrahedral group has the same composition and charge as it has in the ideal formulas for phlogopite and muscovite, and the octahedral layer is neutral, as in the ideal formulas. The 0.67 additional positive charges carried by the 0.67 trivalent cations in the octahedral layer are neutralized by the 0.66 negative charges associated with the 0.33 unoccupied octahedral sites.

Thus in Winchell's formulas for eastonite and siderophyllite, and in Holzner's formula for biotite, trivalent octahedral cations are accommodated in two quite different ways. In one, trivalent cations proxy for bivalent cations in a 1:1 ratio, all the octahedral cationic positions are occupied, and all the additional positive charges carried by the proxying trivalent cations form a positive charge on the octahedral layer and are neutralized by an equivalent increase in the negative tetrahedral charge due to greater proxying of Al for Si. These relations are expressed in the following equation,

$$n\mathrm{R}^{+3}(\mathrm{VI}) + n\mathrm{R}^{+3}(\mathrm{IV}) = n\mathrm{R}^{+2} + n\mathrm{Si}^{+4}.$$

In the other way of accommodating octahedral cations, the proxying of trivalent for bivalent cations is in a 2:3 ratio. There is, consequently, no increase in the total number of positive charges present in the octahedral layer, nor is there any change in the layer charge relations, nor in the composition of the tetrahedral layers as compared to the ideal formulas for muscovite and phlogopite, but there is a decrease in the number of octahedral sites occupied equivalent to one-half the number of octahedral trivalent cations. These relations are expressed in the following equation,

$$2n\mathrm{R}^{+3}(\mathrm{VI}) = 3n\mathrm{R}^{+2} + n \textit{ Oct. Sites}.$$

In the first type of accommodation for trivalent octahedral cations the formula is characterized by full octahedral occupancy, a positive octahedral charge, and a negative tetrahedral charge greater than 1.00 by the number of trivalent octahedral cations; in the second type of accommodation the formula is characterized by deficient octahedral occupancy, a neutral octahedral layer, and a negative tetrahedral charge close to 1.00.

Structural formulas calculated for more than 200 published analyses of phlogopites, biotites, siderophyllites, and lepidomelanes, show that few natural trioctahedral micas accommodate the additional charges carried by trivalent octahedral cations entirely by either one of these ways or the other. Almost all the formulas studied showed the characteristics of both equations. Part of the additional positive charges are neutralized by an increase in negative tetrahedral charge, and a part are neutralized by negative charges associated with unoccupied octahedral positions, as in the following formula,

$$\Big[\underbrace{\overset{+.31}{(\mathrm{Al}_{.35}\mathrm{Fe}^{+3}_{.36}\mathrm{Fe}^{+2}_{1.08}\mathrm{Mg}_{1.01})}}_{2.80}\overset{-1.27}{(\mathrm{Si}_{2.73}\mathrm{Al}_{1.27})}\mathrm{O}_{10}(\mathrm{OH})_2\Big]^{-.96}(\mathrm{K}, \mathrm{Ca}/2)^{+.96}_{.89}.$$

In this formula the octahedral trivalent cations carry 0.71 more positive charge than the same number of divalent cations would carry. Of this additional charge, 0.31 forms a positive charge on the octahedral layer which is approximately neutralized by the increased negative charge on the tetrahedral layer, and the other 0.40 are neutralized by the 0.40 charges associated with the 0.20 unoccupied octahedral sites. The degree to which accommodation for the additional positive charges carried by trivalent (and quadrivalent) cations is made by one method or the other varies greatly. In general, however, there seems to be a tendency to greater accommodation by the first method mentioned above in trioctahedral micas of low octahedral trivalent cation content, with increasing tendency to accommodation by the second method as the octahedral trivalent cation content increases. This is shown in Table 4 by average formulas (calculated from more than 150 analyses) for trioctahedral micas arranged in order of increasing octahedral trivalent (and quadrivalent) cation content. In trioctahedral micas having very low octahedral trivalent cation content, 0.10 sites, for example, all the additional positive charges form a positive charge on the octahedral layer, and are neutralized by an equivalent increase in the negative tetrahedral

TABLE 4. AVERAGE FORMULAS FOR TRIOCTAHEDRAL MICAS, IN ORDER OF INCREASING OCTAHEDRAL TRIVALENT (AND QUADRIVALENT) CATION CONTENT

Octahedral trivalent ion content	Average formula
0.00–0.20	$\underbrace{\overset{+.10}{[(Al_{.05}Fe^{+3}_{.05}Fe^{+2}_{.05}Mg_{2.85})}}_{3.00}\overset{-1.10}{(Si_{2.90}Al_{1.10})}O_{10}(OH)_2]^{-1.00}K^{+1.00}_{1.00}$
0.21–0.40	$\underbrace{\overset{+.15}{[(Al_{.15}Fe^{+3}_{.10}Fe^{+2}_{.35}Mg_{2.35})}}_{2.95}\overset{-1.15}{(Si_{2.85}Al_{1.15})}O_{10}(OH)_2]^{-1.00}K^{+1.00}_{1.00}$
0.41–0.60	$\underbrace{\overset{+.20}{[(Al_{.10}Ti_{.10}Fe^{+3}_{.20}Fe^{+2}_{.90}Mg_{1.55})}}_{2.85}\overset{-1.20}{(Si_{2.80}Al_{1.20})}O_{10}(OH)_2]^{-1.00}K^{+1.00}_{1.00}$
0.61–0.80	$\underbrace{\overset{+.25}{[(Al_{.20}Ti_{.15}Fe^{+3}_{.25}Fe^{+2}_{1.25}Mg_{.90})}}_{2.75}\overset{-1.25}{(Si_{2.7}.Al_{1.25}}O_{10}(OH)_2]^{-1.00}K^{+1.00}_{1.00}$
0.81–1.00	$\underbrace{\overset{+.25}{[(Al_{.25}Ti_{.15}Fe^{+3}_{.30}Fe^{+3}_{1.25}Mg_{.75})}}_{2.70}\overset{-1.25}{(Si_{2.75}Al_{1.25})}O_{10}(OH)_2]^{-1.00}K^{+1.00}_{1.00}$

charge, and there is full octahedral occupancy, as shown in the first formula. In the second average formula 0.15 of the 0.25 additional positive charges carried by trivalent octahedral cations form a positive charge on the layer, and are neutralized by an equivalent increase in the negative tetrahedral charge and 0.10 are neutralized by the negative charges associated with 0.05 unoccupied octahedral sites. In the third formula two-fifths of the 0.50 additional positive charges are neutralized by the first method and three-fifths by the second method. And in the fifth formula 0.60 of the 0.85 additional positive charges are neutralized by the second method, and only 0.25 by the first method. It must be understood, of course, that in some of these micas, whatever their octahedral trivalent cation content, adjustment by one method or the other is emphasized and these average formulas show only the average trend.

Because of the neutralization of some of the additional positive charges carried by trivalent (and quadrivalent) octahedral cations by negative charges associated with unoccupied octahedral sites, octahedral occupancy in most trioctahedral micas is somewhat less than 3.00 sites. Thus they are not truly trioctahedral, nor are they truly octaphyllites, as they generally have fewer than 8 cations for 12 anions.

Replacement by univalent cations

The ferrous lithium micas, protolithionites and zinnwaldites, are, like siderophyllite, characterized by high Fe^{+2} and very low Mg content. These similarities in compositional characteristics suggest siderophyllite as the prototype from which the ferrous lithium micas are derived, just as muscovite can be considered the prototype from which the lithian muscovites are derived. Starting with siderophyllite, formulas calculated from published analyses of protolithionite and zinnwaldite show a general increase in Si, and a general decrease in Al(IV) and Fe^{+2} with increase in Li. The relative amounts of Al(VI) and Fe^{+3} vary considerably but the total amount of octahedral R^{+3} cations remains quite constant. This is illustrated by the analyses shown in Table 5 and by the formulas calculated from these analyses shown in Table 6.

Li replacing Fe^{+2} in a 2:1 ratio in an isomorphous series of this sort would cause no change in the layer-charge relations but would increase octahedral occupancy by an amount equal to one-half the Li added. The gradual change in the layer charges in these formulas from siderophyllite to cryophyllite, from an octahedral charge of $+0.25$, with a tetrahedral charge of -1.32, to an octahedral charge of -0.42, indicates that Li has replaced Fe^{+2} in a less than 2:1 ratio. The ratio varies from one formula to the next, but the overall Li:Fe^{+2} ratio is 1.4:1. Thus the proxying Li cations carry fewer positive charges than the Fe^{+2} cations they proxy,

Table 5. Analyses of Siderophyllites, and Ferrous Lithium Micas Showing Change in Composition with Increase in Li_2O Content

	1	2	3	4	5	6
SiO_2	32.52	38.00	41.78	45.87	48.40	52.17
Al_2O_3	19.07	27.00	22.76	22.50	21.62	16.39
Fe_2O_3	10.08	—	.98	.66	.24	4.11
FeO	17.83	18.50	14.24	11.66	7.19	5.99
MgO	.70	.12	.55	—	—	Tr.
MnO	.21	.64	—	1.75	1.97	.32
Li_2O	.39	1.44	2.42	3.28	4.18	4.99
CaO	.73	1.30	—	—	.06	Trace
Na_2O	.21	1.37	.67	.42	1.12	.63
K_2O	8.53	8.15	10.51	10.46	10.45	10.48
TiO_2	.21	.64	—	—		
H_2O^+	3.17	3.00	1.41	.91	.64	1.46
H_2O^-	3.21	.04			.24	
F	2.36	1.66	6.48	7.94	2.24	7.02
	99.22	101.86	101.80	105.53	100.83	103.56
O=F	.99	.70	2.72	3.33	1.04	2.95
Total	98.23	101.16	99.08	102.20	99.79	100.61

1. Ebisu mine, Naegi District, Japan; Shibata, 1952, p. 162, no. 6.
2. Volhynia, Russia; Tsyganov, 1954, p. 386, no. 9.
3. Altenburg, Saxony, Germany; Kunitz, 1924, p. 413.
4. Zinnwald, Erzgebirge, Bohemia; Dana, 1892, p. 626, no. 1. Total includes 0.08 per cent P_2O_5.
5. Lingwu, Hunan, China; Meng and Chang, 1935, p. 56. Total includes 2.48 per cent B_2O_3.
6. Cape Ann, Rockfort, Mass.; Clarke, 1886, p. 358, B.

and the octahedral layer becomes increasingly deficient in positive charges, necessitating changes in the charge and composition of the tetrahedral layers, increase in Si and decrease in Al(IV).

Cryophyllite has the lithium content of a lepidolite and may be termed a "ferroan" lepidolite. Further replacement of Fe^{+2} by Li in the ratio found in these formulas produces a formula close to polylithionite, with some Fe^{+3} proxying for Al(VI).

Thus lepidolites may be interpreted, compositionally, as if derived from muscovite, by substitution of Li for Al(VI), or from siderophyllite, by substitution of Li for Fe^{+2}. In either substitution the substitution ratio is such that the substituting Li cations carry fewer positive charges than the cations replaced, with the result that increase in Li content is accompanied not only by decrease in Al(VI) or Fe^{+2}, as the case may be,

Table 6. Formulas Calculated from Analyses Given in Table 5, Showing Change in Composition and in Layer Charge Relationship with Increase in Li Content

	Formula	% Li_2O
1. Siderophyllite	$[\overset{+.25}{\underbrace{(Al_{.52}Ti_{.01}Fe^{+3}_{.62}Fe^{+2}_{1.22}Mg_{.09}Mn^{+2}_{.02}Li_{.13})}_{2.61}}\overset{-1.32}{(Si_{2.68}Al_{1.32})}O_{10}(OH)_2]^{-1.07}K(Na, Ca/2)^{+1.06}_{.99}$	0.39
2. Protolithionite	$[\overset{+.06}{\underbrace{(Al_{1.06}Ti_{.03}Fe^{+2}_{1.12}Mn^{+2}_{.04}Mg_{.01}Li_{.42})}_{2.68}}\overset{-1.24}{(Si_{2.76}Al_{1.24})}O_{10}(OH)_2]^{-1.18}K(Na, Ca/2)^{+1.15}_{1.04}$	1.44
3. Protolithionite	$[\overset{-.15}{\underbrace{(Al_{1.04}Fe^{+3}_{.05}Fe^{+2}_{.87}Mg_{.06}Li_{.71})}_{2.73}}\overset{-.93}{(Si_{3.07}Al_{.93})}O_{10}(OH)_2]^{-1.08}K(Na)^{+1.08}_{1.08}$	2.42
4. Zinnwaldite	$[\overset{-.16}{\underbrace{Al_{1.08}Fe^{+3}_{.04}Fe^{+2}_{.68}Mn^{+2}_{.10}Li_{.92})}_{2.82}}\overset{-.79}{(Si_{3.21}Al_{.79})}O_{10}(OH)_2]^{-.95}K(Na)^{+.94}_{.94}$	3.28
5. Zinnwaldite	$[\overset{-.40}{\underbrace{(Al_{1.11}Fe^{+3}_{.01}Fe^{+2}_{.42}Mn^{+2}_{.12}Li_{1.16})}_{2.82}}\overset{-.65}{(Si_{3.35}Al_{.65})}O_{10}(OH)_2]^{-1.05}K(Na)^{+1.07}_{1.07}$	4.18
6. Cryophyllite	$[\overset{-.57}{\underbrace{(Al_{.90}Fe^{+3}_{.21}Fe^{+2}_{.34}Mn^{+2}_{.02}Li_{1.38})}_{2.85}}\overset{-.42}{(Si_{3.58}Al_{.42})}O_{10}(OH)_2]^{-.99}K(Na)^{+1.00}_{1.00}$	4.99

Table 7. Analyses of Lepidolites Showing Change in Composition with Increase in Li_2O Content

	1	2	3
SiO_2	49.19	51.07	57.03
Al_2O_3	24.81	22.05	15.55
FeO	0.24[a]	0.23[a]	0.12[a]
MgO	0.05	0.09	0.22
MnO	2.51	0.76	0.72
Li_2O	5.10	5.89	6.84
CaO	Trace	Trace	Trace
Na_2O	0.52	0.82	0.44
K_2O	10.25	9.70	10.65
Rb_2O	1.78	1.38	1.35
Cs_2O	0.19	0.09	0.40
TiO_2	0.08	0.06	0.03
H_2O^-	1.21	—	0.49
H_2O^+		0.96	0.74
F	6.89	7.13	9.00
	102.82	100.23	103.72[b]
O=F	2.90	3.00	3.79
	99.92	97.23	99.93

[a] Total Fe reported as FeO.

[b] Includes 0.14 per cent Nb_2O_5.

1. Ohio City, Colo.; Winchell, 1942, p. 115, no. 10, 6-layer lepidolite structure.
2. Calgoorie, Western Australia; Stevens, 1938, p. 615, no. 14. 6-layer hexagonal lepidolite structure.
3. Antsongombato, Madagascar, Stevens, 1938, p. 615, no. 16. 1-layer lepidolite structure. Total includes 0.14 per cent Nb_2O_5.

but also by decrease in Al(IV), and by increase in Si and in the number of octahedral positions occupied, or, otherwise expressed, increase in Li content is accompanied by increase in negative octahedral charge and by decrease in negative tetrahedral charge as illustrated in the following formulas calculated from the analyses in Table 7.

1. $$\Big[\underbrace{\overset{-.35}{(Al_{1.32}\overset{+2}{Fe}_{.01}\overset{+2}{Mn}_{.14}Li_{1.39})}}_{2.86}\overset{-.67}{(Si_{3.33}Al_{.67})}O_{10}(OH)_2\Big]^{-1.02}K(Na, Rb, Cs)^{+1.03}_{1.03}$$

2. $$\Big[\underbrace{\overset{-.52}{(Al_{1.25}\overset{+2}{Fe}_{.01}Mg_{.01}\overset{+2}{Mn}_{.04}Li_{1.61})}}_{2.92}\overset{-.52}{(Si_{3.48}Al_{.52})}O_{10}(OH)_2\Big]^{-1.04}K(Na, Rb, Cs)^{+1.02}_{1.02}$$

3. $$\Big[\underbrace{\overset{-.87}{(Al_{1.05}\overset{+2}{Fe}_{.01}Mg_{.02}\overset{+2}{Mn}_{.04}Li_{1.84})}}_{2.96}\overset{-.17}{(Si_{3.83}Al_{.17})}O_{10}(OH)_2\Big]^{-1.04}K(Na, Rb, Cs)^{+1.04}_{1.04}$$

The general trend is, therefore, toward an end member in which the entire composite layer charge is on the octahedral layer, and in which the tetrahedral layers are completely occupied by Si and neutral, as in the hypothetical end member polylithionite,

$$\left[\overset{-1.00}{(\mathrm{Al}_{1.00}\mathrm{Li}_{2.00})}\overset{.00}{\mathrm{Si}_{4.00}}\mathrm{O}_{10}(\mathrm{OH})_2\right]^{-1.00}\mathrm{K}^{+1.00}_{1.00}$$

In the few analyses of lithian biotites available, Li_2O does not exceed 1.5 per cent, equivalent to about 1.5 formula sites. The data are so meager as to permit no conclusions as to replacement ratios or layer charge relations.

CONCLUSION

A study of formulas calculated from published analyses of dioctahedral and trioctahedral micas shows that substitution of octahedral cations having a different valence from the cation ideally present produces changes in the octahedral-layer charge which is accompanied by layer charge and compositional adjustments. If the substituting cation carries less charge than the cation substituted, the deficiency in charge is not entirely compensated by increase in the number of cations substituted, the octahedral anions are not entirely neutralized, and the octahedral layer has a negative charge. This is accompanied by decrease in the negative charge on the tetrahedral layers, that is, increase in Si and decrease in tetrahedral Al. In such micas, as in most dioctahedral and in lithium micas, Si occupies more than 3.00 formula sites and tetrahedral Al less than 1.00 formula sites. The total composite layer is the sum of two negative charges.

If the substituting cation has a higher charge than the octahedral cation substituted, the excess in charge is seldom entirely compensated by decrease in the number of cations substituted, and there are not enough octahedral anions to neutralize all the positive charges present. Thus the octahedral layer has a positive charge, and the tetrahedral layer has a negative charge greater than 1.00 by about an equivalent amount, with a lower Si content and a higher Al(IV) content than in the ideal formula. In such micas, phlogopites, biotites, siderophyllites and lepidomelanes, Si occupies fewer than 3.00 formula sites and Al(IV) occupies more than 1.00. The total layer charge is the algebraic sum of a positive and a negative charge.

As most natural dioctahedral and trioctahedral micas exhibit such substitutions, they differ in layer charge relations and in layer composition from the ideal formulas for such micas to a greater or lesser degree, depending on the degree and ratio of substitution.

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MEMORIAL OF PEGGY-KAY HAMILTON

PAUL F. KERR, *Columbia University, New York, New York*

Miss Peggy-Kay Hamilton, Research Associate in Mineralogy, in the Department of Geology, Columbia University, passed away on September 19, 1959, at the age of 37. Death came suddenly as a result of a brief illness, followed by a cerebral operation for cancer.

Peggy-Kay, as she was known to a large group of graduate students at Columbia University, was a friend, a research adviser, and a constant source of professional information on the solution of mineralogical problems. Technically, she held the post of Research Associate, and was also an assistant on a research project under the joint auspices of the University and the Division of Research of the Atomic Energy Commission.

PEGGY-KAY HAMILTON
1922–1959

Actually, she performed the function of a research professor. Few of the graduate students specializing in mineralogy in the last decade have failed to profit from her guidance. Every thesis in mineralogy received the benefit of her informal review before it was submitted.

Peggy-Kay graduated from Vassar College in April, 1944. and entered Columbia in February, 1945, when she undertook graduate work in geology and mineralogy. She received a Master's Degree in June, 1947, and was well qualified to continue for the Doctorate. She preferred, however, to continue as a full time research assistant, devoting her whole effort to various research problems as they arose. Over the years she completed studies equivalent to several doctorates, but could never be persuaded to complete a thesis and undertake the necessary schedule of examinations. Peggy-Kay's first major research effort was devoted to the American Petroleum Institute study of reference clay minerals, known as Research Project 49. The glossary of clay minerals published by the Institute to a large degree represents her work. She also participated in optical and x-ray studies likewise published, and carried a major portion of the editorial work of the project.

With the development of interest in the mineralogy and occurrence of uranium, Peggy-Kay became involved as a full time research investigator in the studies on the origin and nature of uranium minerals. In this work she participated in eleven brochures published by the Division of Raw Materials of the Atomic Energy Commission. Also, she cooperated or assisted materially in several papers that have been published in the American Mineralogist and the Bulletin of the Geological Society of America. At the time of her death, a paper on the modes of umohoite, largely the result of her investigation, had been accepted by the editor of the American Mineralogist for publication.

Those members of the profession who were fortunate enough to know Peggy-Kay respected her for her constant ladylike behavior, her alert interest in mineralogical research, her friendly willingness to cooperate in the solution of mineralogical problems, and her high standard of professional excellence. She was a member of Sigma Xi, and wore a key which she prized highly. Her competence as a mineralogist was well emphasized by the ease with which she was elected a fellow of the Mineralogical Society of America, and likewise a fellow of the Geological Society of America. She was a regular contributor of abstracts for the Mineralogical Society of America.

Peggy-Kay could have occupied more important positions, and on several occasions was offered posts of considerable professional responsibility at double the salary she was receiving. However, she preferred to remain in New York City, where she was able to live at home with a family she highly cherished. With the death of Peggy-Kay Hamilton, the profession suffers, not only the immediate loss, but many fruitful years of scientific endeavor which would have normally been expected from one who had made such a brilliant start. It is doubtful whether any staff member at Columbia has been more keenly missed on the part of present and former students than Peggy-Kay Hamilton.

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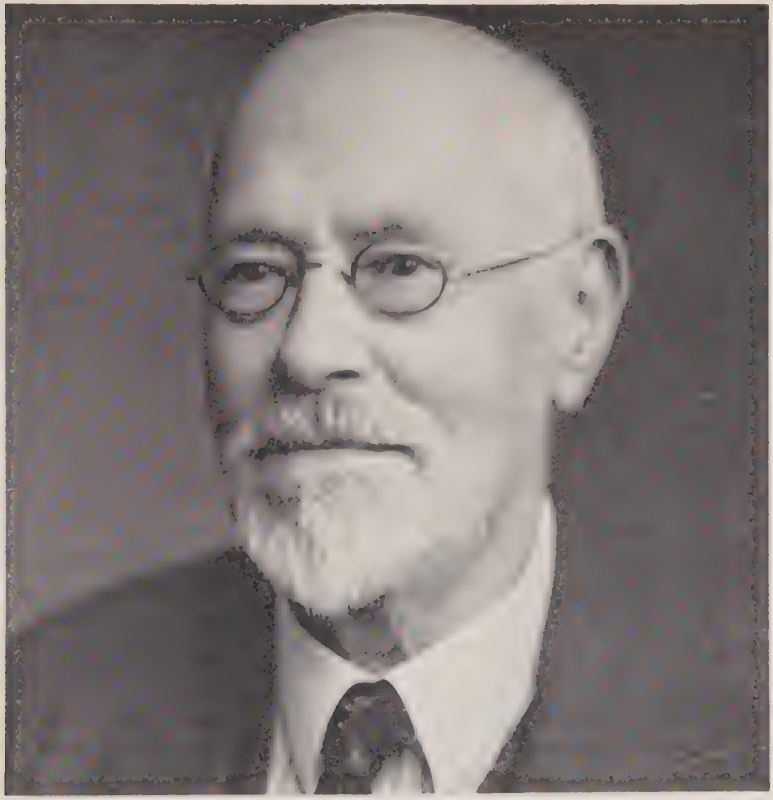
THE AMERICAN MINERALOGIST, VOL. 45, MARCH-APRIL, 1960

MEMORIAL OF LEONARD JAMES SPENCER

C. E. Tilley, *Cambridge, England.*

Leonard James Spencer, Honorary Fellow of the Society and Roebling Medallist, died in London on April 14, 1959 at the advanced age of 88. He was fourth in a line of distinguished Keepers of Minerals in the British Museum, his predecessors being Story-Maskelyne appointed first Keeper in 1857, Sir Lazarus Fletcher and G. T. Prior. All three were Fellows of the Royal Society to which Spencer also was elected in 1925. He was born at Worcester on July 7, 1870, the eldest son of James Spencer, schoolmaster, and Elizabeth Bonser, and was educated at Bradford Technical College (1883–1886) where his father was for many years headmaster of the day school department. With a Royal Exhibition Spencer proceeded to the Royal College of Science for Ireland in Dublin (1886-



Leonard James Spencer
1870–1959

1889) and later entered Cambridge University as a scholar of Sidney Sussex College. Here he studied geology, mineralogy and chemistry gaining a first class in both parts of the Natural Sciences Tripos and was awarded the Harkness scholarship for Geology (1893). It was fortunate for mineralogy that just at the end of his Cambridge course there happened to be one of the infrequent vacancies in the scientific staff of the Mineral Department of the British Museum, and for this post Spencer was the successful candidate. The latter part of the year 1893 he spent at Munich studying under Groth, Weinschenk, and Muthmann. His appointment at the Museum dated from January 1, 1894. Through more than forty years Spencer held office in the Mineral Department, succeeding to the Keepership on the retirement of Prior, but intimate connection with the department lasted for another twenty-four years to the time of his death. He married in 1899 Edith Mary Close (died 1954) and leaves one son and two daughters.

Spencer's achievements fall into three main fields, his work as curator, as an original investigator, and as editor and bibliographer. He was a born collector and an indefatigable and incessant worker and as curator of the natural collections in his department his labors were remarkable for their thoroughness and accuracy. He established in the Mineral Department the system of registration, labelling and cataloguing and it is mainly through his efforts that this collection is now probably the best documented and indexed in the world. His output of original research was considerable and his publications, more than 150 in all, range over a wide field of descriptive mineralogy. Many of his early papers stemmed from the curatorial work he did on the crystallographic catalogue. He described as new the minerals miersite, tarbuttite, parahopeite, chloroxiphite, diaboleite, schultenite, aramayoite and bismutotantalite, besides supplying much new data on incompletely described species. In later years his early interest in meteorites was renewed and greatly stimulated by his visit to South-West-Africa in 1929 where he studied the meteorites of Gibeon and Hoba.

The discovery of silica-glass fragments at meteorite craters (Henbury and Wabar) led him eventually to take up the problem of tektites and their origin. These he believed were the products of the impact of large meteorites on the Earth. It was about this time (1934) on the eve of his retirement that his enthusiasm led him to accept an invitation to join an expedition to the Libyan desert to study the remarkable masses of silica-glass found by P. A. Clayton in 1932. He failed however to find the hoped-for evidence of meteorite craters and returned with the problem of the origin of the glass still unsolved. Spencer's lively interest in meteorites endured to the end, his last publication being a communication on the subject in *Nature* of March 1958.

The third field of Spencer's activity was as editor, bibliographer and abstractor, and in this domain he gave immense service to his science. His work as abstractor began soon after his appointment to the British Museum. From the first he contributed abstracts to the *Mineralogical Magazine* and as early as 1895 he was preparing abstracts in the field of mineral chemistry for the *Journal of the Chemical Society;* later he contributed to the Annual Reports of the same Society. He took over the editorship of the *Mineralogical Magazine* from Miers at the close of 1900 and continued to edit it until the end of 1955. He prepared the indexes of the Magazine covering in all twenty volumes and these appeared in two issues (1895, 1926). He supplied triennially lists of new mineral names (21 lists in all), and obituary notices of mineralogists (eight series) and three supplementary lists of British minerals. In 1920 the Mineralogical Society commenced publication of *Mineralogical Abstracts* with Spencer as editor. These appeared as a sequel to the International Catalogue of Scientific Literature which terminated in 1914. During this period as editor (1920–1955) twelve volumes were issued and he lived to see *Mineralogical Abstracts* appear in a new format as a joint publication of the British and American Mineralogical Societies aiming at a still wider international coverage of mineralogy. The Mineralogical Society celebrated Spencer's fifty years as editor of the Magazine by the issue of a Jubilee number in 1950 to which colleagues at home and abroad contributed papers and on the occasion of the anniversary meeting in November 1950, a dinner was given in his honor.

The last decade of Spencer's service at the British Museum brought him honors and promotion richly deserved. He was elected a Fellow of the Royal Society (1925) and correspondent of the Mineralogical Society of America (1926) and he succeeded to the Keepership of the Mineral Department in 1927. The Roebling Medal was awarded to him in 1940. He served as President of the Mineralogical Society (1936–1939) and became its Foreign Secretary in 1949. Spencer continued an active member of the Society to the end attending regularly meetings of the Council and of the Society itself. He continued also to serve as a member of his successor's team of abstractors for *Mineralogical Abstracts*. He had been a member of the Society for 65 years and for long an honored and familiar figure on the front bench at its meetings. As we look back on his achievements, the unique service he gave to mineralogy, we realize the magnitude of the loss we have sustained. In the annals of the Society he served so well, his passing marks indeed the end of an epoch.

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PRESENTATION OF THE ROEBLING MEDAL TO FELIX MACHATSCHKI

IAN CAMPBELL, *California Institute of Technology and California State Division of Mines*

President Grim, Fellows and Members of the Mineralogical Society, and Guests:

You will recall that tenet of Anglo-Saxon law which holds that a man shall not be called upon twice to place himself in jeopardy for the same crime. Old as it is, this law seems not to have been known to the presidents of the Mineralogical Society, for twice I have been appointed to serve as chairman of Roebling Medal Award committees. I do not mean to imply that—whatever jeopardy may be involved—the chairmanship of the Roebling Medal committee constitutes a crime! Quite the contrary; it is a very great privilege. I make these introductory remarks principally in order to point up the gravity of the problem faced by all Roebling Medal committees; and because this permits me to recall that a decade ago I served as chairman of a Roebling Medal committee which made no award. This was not because there were no men of Roebling Medal stature in the mineralogical world ten years ago, but rather because it seemed to our committee virtually impossible to pick out one who stood sufficiently high above the others to warrant selection. And, more importantly, we were considering a selection in the shadow, so to speak of the preceding medalist, Normal L. Bowen. To find anyone to measure up to our great—and greatly beloved—Roebling Medalist of 1950 was indeed an impossible task.

These problems did not beset the current committee, composed of C. A. Anderson, J. R. Goldsmith, C. E. Tilley and myself. Our very first survey recognized a number of distinguished mineralogists and petrologists, and succeeding discussion rapidly narrowed the field to a very few men distinctly of Roebling Medal calibre. It must be confessed that the final selection, amongst the three or four mineralogists and petrologists whom we regarded as "tops" was not easy; but it was unanimous and was accomplished with the happy thought that mineralogists and petrologists are traditionally long-lived, and that those who were not nominated in 1958 would certainly provide top material for future Roebling Medal committees to consider.

Our 1959 Roebling Medalist, Professor Felix Machatschki, was born in Arnfels, Austria, 64 years ago. He received the Ph.D. degree from the University of Graz in 1922 and until 1927 remained at Graz, first as assistant, later as docent. From 1927 to 1930, he was a visiting lecturer at Oslo, at Manchester and at Göttingen. In 1930 he was called to Tübin-

Felix Machatschki

gen as Professor of Mineralogy and Director of the Mineralogical Institute. There he remained until after the outbreak of the war. From 1941 to 1943 he was at Munich; and in 1943 he went to Vienna, first to occupy the chair already made famous by Tchermak and Becke and later to occupy the chair formerly held by the equally famous Doelter.

We honor Professor Machatschki today for many things, but chiefly for his outstanding contribution to our knowledge of silicate structure. Thirty years ago, we did not know the phrase "scientific break-through"; but with the advantage that hindsight confers, we now recognize—what we did not fully then—that the paper Professor Machatschki published in the *Centralblatt für Mineralogie* in 1928, on "Zur Frage der Struktur und Konstitution der Feldspate," constituted a "real scientific break-through" in the field of mineralogy and crystallography. It was in this paper that we first find the important suggestion that the feldspar structures are based on frameworks of linked SiO_4 and AlO_4 tetrahedra with cations in the interstices. Professor Machatschki suggested also that the difference between the orthoclase and plagioclase feldspars might be due to the fact that the former contains large cations, and the latter small cations. Subsequent analyses have amply confirmed the correctness of these predictions, and the predictions have enabled students of silicates in general and of feldspars in particular to move forward in a way that has been greatly rewarding.

To be sure, even before this paper had been published, Professor Machatschki had communicated his ideas to other workers in the field, and had communicated them so effectively that, I am told, Professor Goldschmidt thenceforth, in his introductory lecture on silicates to his students at Oslo, invariably began with the remark. "So wie jeder Muselmann zu seiner Glückseligkeit vier Frauen braucht, wie Machatschki sagt, so braucht auch jedes Siliziumatom in seiner nächsten Umgebung vier Sauerstoffatome, wie Machatschki sagt!"

Among his many other contributions to our science, of which time permits us here to cite only a few, should be mentioned his classification of and improvements in the notation of silicate structures; his recognition of the possibility of replacement of Si by other ions, as in berzeliite; his studies of the structure of danburite, of dyscrasite, of the epidote group, of the chlorites and of the amphiboles and pyroxenes; his discussion of the problems of isomorphism and isomorphous replacement in relation to atomic radii and structure type; and his interest in relating crystal structure to paragenesis. In addition to his many papers, he is the author of three books: "Grundlagen der allgemeiner Mineralogie und Kristallchemie" (1946), "Vorrate und Verteilung der mineralische Rohstoffe" (1948); and "Spezielle Mineralogie auf geochemischer Grundlage" (1950). Two of these volumes, it deserves to be noted, were brought out in the very difficult years immediately following the war.

Not the least of Professor Machatschki's contributions to our science stem from his role as teacher. First in Tübingen and, in recent years at

Vienna, he has attracted a notable and notably cosmopolitan group of young scholars to his laboratory—from France, from Turkey, from Egypt, from the Scandinavian countries, and from America. All of them have profited from his guidance.

Professor Machatschki was in Vienna at the time of the Russian occupation and did his utmost to preserve the university laboratories and museum from pillage. The reconstruction of facilities and the restoration of this mineralogical center to its former position of world prestige must also be counted among his notable accomplishments. In this connection you must permit me to include here a parapraph from a recent letter from R. C. Evans to my committee colleague, C. E. Tilley, in which Dr. Evans recounts some of his experiences as a member of a technical mission which, immediately after the war, had been sent by the British to visit the principal centers of mineralogic and crystallographic research in central Europe. Dr. Evans remarks: "It was with genuine sorrow that we left Austria. Nowhere in our tour of laboratories from Bonn to Berlin and from Hamburg to Graz had we received such a warm welcome as in Vienna, and nowhere did we meet such a courageous, friendly, and exuberant mineralogist as Felix Machatschki."

Mr. President, it must be abundantly clear, even from this highly condensed review, why the Roebling Medal committee is so happy in their selection, and why it is now for me a high privilege to present to you and to the Society our 1959 Medalist, Dr. Felix Machatschki, scientist, scholar, rare and exuberant spirit, Professor of Mineralogy at the University of Vienna.

THE AMERICAN MINERALOGIST, VOL. 45, MARCH-APRIL, 1960

ACCEPTANCE OF THE ROEBLING MEDAL OF THE MINERALOGICAL SOCIETY OF AMERICA

FELIX K. L. MACHATSCHKI, *University of Vienna, Austria.*

Mr. President, Professor Campbell, Fellows, Members and Guests of the Mineralogical Society of America:

I have two feelings on this third of November, a day so important to me that I would like to call it the day of highest feeling in my life as far as my scientific devotion goes.

First, I feel very proud of the honor to be conferred upon me, the Roebling Medal, which is so highly esteemed all over the world. The second feeling is a more personal one: do I really deserve this unique distinction? Looking around I see so many distinguished mineralogists who would have merited this honor at least as much as I, to say nothing of the many others who are not with us today.

You may be interested in hearing why I started in mineralogy and how I became interested in silicate and other structures. Originally I had not planned to become a mineralogist at all. After the first world war, I began my studies in biology, physics, and mathematics in order to obtain the teacher's degree for high schools. In doing this I also had to study mineralogy, geology, and chemistry, but only at an elementary level. After finishing my examinations I became a teacher of natural history. But it was Rudolf Scharizer, Professor of Mineralogy and Petrology at the University of Graz, well known because of his work on granite pegmatite minerals and on natural and artificial iron sulfates, who soon afterwards insisted that I join his department to become his assistant in teaching classical mineralogy, petrology, and crystallography. He also urged me to do research work in crystallography and mineral and rock analysis. Although Scharizer was an excellent teacher, able to make all phases of classical mineralogy a living subject, I at first did not like this kind of work because I still was more interested in biological problems. I did not yet realize how significant mineralogy and crystallography were becoming, with their growing importance as a means for understanding the rules governing the structure of non-living materials, and as a means for relating geology, physics, and chemistry.

But with time I became especially interested in mineral chemistry. In the course of my own analytical work and by using the publications of S. L. Penfield, W. T. Schaller, R. Mauzelius and many others, and the data collections in Dana's System and Doelter's Handbuch, I soon became disappointed with the various concepts of the molecular constitution of minerals, especially of the silicate minerals. These concepts could not explain in a uniform satisfying manner the variability in composition of many mineral groups with apparently analogous crystallographic and crystal-physical behavior. I became acquainted with the work of V. M. Goldschmidt and his co-workers on the crystal structure of simple compounds. By means of a Rockefeller fellowship I was able to go to the laboratory of Goldschmidt in Oslo. Here I studied the early publications of Sir William L. Bragg and his co-workers on the crystal structure of some silicates. Now it was merely a synthesis of all these results which made it inevitable to abandon the assumption of discrete molecules in the silicates and other groups of inorganic compounds. Instead, the replacement of tetravalent Si by trivalent Al was assumed, as well as other non-isovalent substitutions.

I never intended to give a complete structural classification of the silicates; I only wished to emphasize some principles, e.g., the concept of infinite complex ions in the form of chains and frameworks. Linus Pauling soon after enlarged my proposals by the detecting of infinite tetrahedron sheets in the micas and clay minerals, and B. E. Warren worked out the band structure of the amphiboles. Many other crystallographers in all countries have contributed to this field, working out other types of chains, bands and sheets in the silicate minerals. It soon became clear that the concept of definite and infinite complex ions is not limited to the silicates. Despite the different character in the chemical bonding and despite other coordination numbers, it also holds for other groups of natural and artificial inorganic compounds, e.g., the borates, titanates, niobates, aluminium fluorates, and even hydrates.

Much work has been done in this field, but there is still more to be done, especially in the direction of a refinement which takes into account the thermodynamical conditions of formation, as has been shown in the case of the feldspars by W. H. Taylor, J. R. Goldsmith, Fr. Laves and others.

Referring again to the great honor which you are bestowing upon me, I find it appropriate to recall to our memories how much we owe to Colonel Roebling for his far-sighted and generous attitude, which led him to assist the Mineralogical Society of America and other institutions. This Society has probably become the largest and certainly a worldwide and most highly esteemed Mineralogical Society, with far more than 2000 fellows and members in all countries of the world. Roebling has in this way contributed a great deal to the development of our beloved science in all its branches.

I wish to thank you most sincerely for awarding me the Roebling Medal. You are expressing your appreciation of my very small contribution to our science. Nothing can be more gratifying to a scientist than the knowledge that something he has done is being honored in this fine manner.

THE AMERICAN MINERALOGIST, VOL. 45, MARCH-APRIL, 1960

PRESENTATION OF THE 1959 MINERALOGICAL SOCIETY OF AMERICA AWARD TO HARRY F. W. TAYLOR*

Lloyd W. Staples, *Department of Geology, University of Oregon, Eugene, Oregon.*

Mr. President, Fellows, Members of the Mineralogical Society of America, and Guests:

This is the ninth consecutive year that the Mineralogical Society of America has chosen a recipient for its Award. The task of selection is not easy, as it requires not only that the recipient be a young man, but that the search be world-wide in scope.

It is my very great pleasure to present to you a person who more than adequately fills the stringent Award requirements. Dr. Harry F. W. Taylor, born in Nottingham, comes to us from the University of Aberdeen. It is interesting that the second recipient of the Mineralogical Society of America Award, Dr. Fred H. Stewart, also originally came from Aberdeen, although at the time of the award he was at Durham University in England.

Doctor Taylor's work provides a good example of the interdependence of the sciences. He graduated in chemistry from Nottingham University and did research on the hydrothermal synthesis of aluminosilicates under Professor Barrer. Later he held a post-doctoral fellowship at Birkbeck College in physics under Professor Bernal, where his work on the hydrated calcium silicates started. Moving to Aberdeen, he continued his work on the calcium silicates and became increasingly more interested in the mechanisms of thermal and hydrothermal transformations of silicates.

It is difficult to determine exactly when Doctor Taylor became interested in mineralogy and crystallography, because the development of his interest was dictated by a steadily increasing need in his physicochemical studies. His work on silicates pointed out the advantage of following his studies of synthetic materials with comparative studies of natural materials, which often were better crystallized and, consequently, permitted single crystal x-ray studies. However, in order to make full use of these

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* Harry Francis West Taylor, Department of Chemistry, University of Aberdeen, Old Aberdeen, Scotland. Born in Nottingham, England, Feb. 24, 1923. University College, Nottingham. B.Sc. London External, 1943. Ph.D. London External, 1947. D.Sc. London 1957. Research student and Demonstrator, University College Nottingham, 1943–46; Demonstrator, Bedford College, University of London 1946–48; Research Fellow, Birkbeck College, University of London, 1948–53; Lecturer, Department of Chemistry, University of Aberdeen, 1953 -. Fellow, Chemical Society, London; Member, Mineralogical Society, London.

Harry F. W. Taylor

natural materials, it was necessary to have a clear picture of their structure, and quite often a satisfactory structural study was not available. He solved this problem by making his own crystallographic studies.

It was in 1953, after publishing some 14 papers, mostly in the *Journal* *of the Chemical Society*, that Doctor Taylor presented his first paper to the *Mineralogical Magazine.* In the short time since 1953, he, with some of his colleagues, has described three new minerals and one heretofore unknown mineral analogue. Although the discovery and description of new minerals is always fascinating and rewarding, I consider an even more important contribution to be his clarification of the relations and status of a host of other minerals whose structures he has carefully determined. In ten papers, he has described the structure and relations of many of the hydrous or basic calcium silicates, such as gyrolite, crestmoreite, riversideite, truscottite, jurupaite, xonotlite, afwillite, tobermorite, zeophyllite, and foshagite, indicating identities in certain cases such as between jurupaite and xonotlite. The state of confusion existing with respect to these minerals was as formidable as their names. His sweeping out of ambiguities is a most excellent type of mineralogical housekeeping. Many of his observations were recorded in a monograph published in 1956, with Heller as co-author, and titled: "Crystallographic Data for the Calcium Silicates." This is a valuable contribution which Doctor Taylor is attempting to make obsolete through his subsequent work.

President Grim, in recognition of his outstanding contributions toward an understanding of the calcium silicates, I present Dr. Harry F. W. Taylor to you to receive the Award of the Mineralogical Society of America.

THE AMERICAN MINERALOGIST, VOL. 45, MARCH-APRIL, 1960

ACCEPTANCE OF THE MINERALOGICAL SOCIETY OF AMERICA AWARD

H. F. W. TAYLOR, *Department of Chemistry, The University, Old Aberdeen, Scotland.*

Mr. President, Fellows, Members of the Mineralogical Society of America, and Guests:

I want to begin by saying how glad I am to be in the United States again. I was here previously two years ago, and it is a real pleasure to come here once more.

When I received your Secretary's letter informing me that I had been given this Award, I could hardly believe my good fortune. So many names came to my mind of mineralogists, both in the United States and abroad, whose claims seemed better than mine. However, the Council has made its decision, and I can only express my sincere, and I hope, humble thanks.

Many people, and several institutions, ought to share this honor with me. I am thinking of my teachers, especially Professor Barrer, who first interested me in silicate chemistry and taught me much about it, and Professor Bernal, who taught me something from his great knowledge of crystallography. I am indeed sorry that Dr. L. J. Spencer is no longer alive to hear me say that his friendly criticisms of my first papers in the *Mineralogical Magazine* helped me to become something more like a mineralogist. I did not enjoy rewriting them at the time, but realize now how much he taught me. I have been fortunate in the places where I have worked, especially Birkbeck College, London, and Aberdeen University. Both have provided excellent facilities for research. Perhaps most important of all, I have had some first rate collaborators. On the mineralogical side of my research, Alan Gard, Lesley Dent Glasser, and Roy Buckle in particular have contributed much, both in experimental skill and in interpretation and ideas, for which I am to-day receiving the credit.

I believe that it is good to combine fundamental research with work of a more applied kind. In my case the fundamental work has mostly been on the chemistry and crystallography of calcium silicates, and the applied work on the hydration reactions of cements. I have found that the fundamental work provides a stimulus for the applied work, and vice versa. I am not sure that one cannot go further and suggest that the university scientist should feel a certain moral obligation to devote part of his energy to work with some degree of technical significance, and that industry should feel a corresponding obligation to support work that is other than of immediate utility. I have enjoyed happy relations with industrial firms, both in the United States and in Great Britain. It is fitting that I acknowledge here my gratitude to industry in the United States in particular for both the ideas and the financial support that it has provided.

As Professor Staples has told you, I was trained as a chemist, and am to-day a lecturer in inorganic chemistry. There was a time, during the last century, when inorganic chemistry was often called mineral chemistry, and was closely allied with mineralogy. In recent years this link has become somewhat tenuous, and the emphasis in inorganic chemistry has moved towards finite groups of atoms—coordination chemistry, problems of the chemical bond, and the like. I do not wish in the least to belittle the importance of these fields of study. However, I believe most strongly that a renewed closer association with mineralogy would benefit both disciplines.

I have tried, both as a research worker and as a teacher, to do something to make the links between the two subjects a little stronger. I hope that, in future years, many new links will be forged in different laboratories, and that I may play my part in this process. In that way, I might show myself worthy of the honor that you have given me.

THE AMERICAN MINERALOGIST, VOL. 45, MARCH–APRIL, 1960

PROCEEDINGS OF THE FORTIETH ANNUAL MEETING OF THE MINERALOGICAL SOCIETY OF AMERICA AT PITTSBURGH, PENNSYLVANIA

C. S. Hurlbut, Jr., *Secretary*

The fortieth meeting of the Society was held on November 2–4, 1959 at the Penn-Sheraton Hotel, Pittsburgh, Pennsylvania. The scientific sessions were integrated with those of the Geological Society of America and other affiliated Societies. A total of thirty-five scientific sessions were held; of these two were devoted to mineralogy, two to petrology, one to crystallography and five to geochemistry, at which 107 papers were presented.

The annual luncheon of the Society on November 3 was attended by 234 fellows, members and guests. This exceeded by two the number attending the luncheon in Atlantic City in 1957 and was thus the largest number ever to attend a Society luncheon. Following the luncheon the seventeenth presentation of the Roebling Medal was made to Felix Machatschki and the ninth presentation of the Mineralogical Society of America Award was made to Harry F. W. Taylor.

On the afternoon of November 3 the Society was addressed by the Retiring President, Ralph E. Grim, on the subject, *Some Applications of Clay Mineralogy*. The annual business meeting was held at 4:30 on the afternoon of November 3, at which brief reports were given by the Secretary, Treasurer and Editor. It was voted in response to a motion made by Professor C. W. Wolfe That the Society spread upon its records an expression of appreciation of the services rendered the Society by Professor C. S. Hurlbut, Jr. during his fifteen years as Secretary.

Council Action

The 1959 Council of the Society met on the afternoon and evening of November 1 for a total of six hours and discussed 23 items of business. Although there were the usual number of items, the time needed to consider them was less than in many years. This was because of the excellence of the reports submitted by the several committees that had been at work throughout the year.

At its first meeting, in November 1958, the 1959 Council appointed a Policy Committee to consider the future financial needs of the Society and to make recommendations to the Council at its Pittsburgh meeting. Council action on certain aspects of this Committee report affects all members and fellows of the Society and is thus given below.

Publication costs of the *American Mineralogist* as well as other Society expenses have been rising continuously during the past decade while dues have remained constant. At present neither the dues of Members and Fellows nor the subscription rate of the *American Mineralogist* is sufficient to meet the publication costs of the journal. Acting on recommendations of the Policy Committee the Council voted the following:

Subscription to the American Mineralogist. That the subscription rate of the *American Mineralogist* be $10.00 a year beginning in 1961.

Dues of Members and Fellows. That steps be taken to raise the dues of members and fellows to $8.00 a year.

Student Members. That the dues of members who are students be set at one-half the dues of other members.

Mineralogical Abstracts to Members and Fellows. That, if dues are raised to $8.00 a year, Mineralogical Abstracts be made available to members and fellows for $2.00 a year additional. Note. Students paying the reduced rate will pay $3.00 for Mineralogical Abstracts.

Change in the By-Laws. In order to effect the increase of dues of members and fellows, the Council recommends the following changes in Article II, Sections 1 and 2 of the by-laws:

From

Section 1. The annual dues for members shall be four dollars ($4) payable in January.
Section 2. No person shall be accepted as a fellow of the Mineralogical Society of America unless he pays dues for the year within three months after notification of his election. The annual dues for fellows shall be five dollars ($5) payable in January.

To

Section 1. The annual dues for members and fellows shall be eight dollars ($8) payable in January. Members who are students shall pay one-half the dues of other members.
Section 2. No person shall be accepted as a fellow of the Mineralogical Society of America unless he pays dues for the year within three months after notification of his election.

The Council also recommends the following change in Article II, Section 3 of the by-laws.

From

Section 3. Fellows who have reached the age of seventy years, and who have paid dues for at least thirty (30) years, shall be exempt from further payment of dues.

To

Section 3. Fellows and *members* who have reached the age of seventy years, and who have paid dues for at least thirty (30) years, shall be exempt from further payment of dues.

The Council requests that the general membership of the Society be asked to vote on the above recommended changes in Article II of the by-laws.

International Mineralogical Association. A report was given the Council by Professor C. Frondel, delegate to the International Mineralogical Association Congress held in Zurich, Switzerland August 28–September 4, 1959. A summary of this report was printed in the *American Mineralogist* **44,** 1319 (1959). Another meeting of the International Mineralogical Association is to be held in Copenhagen, Denmark in 1960 during the meetings of the XXI International Geological Congress.

Honorary Fellows. The Council voted that Felix Machatschki, the 1959 recipient of the Roebling Medal, be made an Honorary Fellow.

ACTION BY THE 1960 COUNCIL

The 1960 Council met on November 4, 1959 for three hours and discussed fourteen items of business.

International Mineralogical Association. The Council voted that $150.00 be paid during 1960 as the Society's dues to the International Mineralogical Association.

Advertising in the American Mineralogist. It was agreed by the Council that it would be desirable to increase the amount of advertising carried in *American Mineralogist* in order to increase the Society's revenue. The Council voted that Martin L. Ehrmann be appointed to carry out this project.

Editor of the American Mineralogist. The Council voted that Lewis S. Ramsdell be appointed Editor of the *American Mineralogist* for the year 1960.

Articles for the Amateur. The 1958 Council appointed Richard H. Jahns to arrange for occasional articles in the *American Mineralogist* of interest to non-professional mineralogists. The 1960 Council voted that Dr. Jahns be authorized to continue with this activity.

Endowment Committee. The Council voted that this committee be reactivated.

Commercial Audit of the Society Books. Upon recommendation of the 1959 Auditing Committee, the Council voted that the Financial Advisory Committee study the possibility of having a commercial firm audit the Society books and make specific recommendations to the Council in November 1960.

Committee Appointments. President Murdoch made the following committee appointments, which were approved by the Council.

1960 Committees of the Mineralogical Society of America

Nominating Committee for Fellows

R. Roy, *Chairman*	1960–63
C. O. Hutton	1958–60
A. L. Anderson	1958–60
J. L. Kulp	1959–61
E. N. Cameron	1960–63
J. D. H. Donnay	1960–63

Roebling Medal Committee

J. R. Goldsmith, *Chairman*
A. F. Buddington
E. Ingerson
T. S. Lovering
G. Tunnell
H. Strunz

Program Committee

B. Mason
H. Winchell

Nomenclature Committee

M. Fleischer, *Chairman*	1958–60
C. S. Hurlbut	1958–60
J. V. Smith	1959–61
G. Tunnell	1959–61
A. Pabst	1960–62
G. Faust	1960–62

MSA Award Committee

H. Winchell, *Chairman*
H. F. W. Taylor
J. Verhoogen
W. D. Keller
E. W. Roedder
J. B. Thompson

Financial Advisory Committee

E. P. Henderson, *Chairman*	1959–61
S. Goldich	1959–60
A. Montgomery	1960–63

Nominating Committee for Officers

V. B. Meen, *Chairman*
B. Mason
C. L. Christ
J. L. Gilson
L. Staples

Auditing Committee

G. Kullerud, *Chairman*
A. Van Valkenburg
M. L. Lindberg

Board of Associate Editors

D. J. Fisher	1958–60
G. T. Faust	1958–60
G. W. Brindley	1959–61
A. Pabst	1959–61
E. W. Roedder	1960–62
R. H. Jahns	1960–62

Mineralogical Abstracts

L. B. Berry, *Chairman*
A. Pabst
M. E. Mrose
H. Winchell
K. M. Servos

Advertising

M. L. Ehrmann

Endowment Committee

C. B. Slawson
A. O. Woodford

Representatives

American Geological Institute

Board of Directors:

J. D. Barksdale	1959–60
R. M. Grogan	1960–61

Government Relations Committee:

P. F. Kerr	1959–61

International Mineralogical Association

Representative:
D. J. Fisher

Delegates:

C. S. Hurlbut	Voting
C. Frondel	
J. Murdoch	
A. Pabst	
G. Switzer	
E. W. Heinrich	Alternates
R. E. Grim	

American Association for the Advancement of Science

D. J. Fisher
E. E. Wahlstrom

National Research Council

W. F. Bradley	1960–63

Members of Commissions:

Abstracts —H. Winchell
Data —G. T. Faust
New Minerals—D. McConnell
Museums —G. Switzer

Delegate to XXI International Geological Congress

—J. Murdoch
—R. E. Grim (alternate)

REPORT OF THE SECRETARY

To the Council of the Mineralogical Society of America

Election of Officers and Fellows

Eight hundred and forty-four ballots were cast in the election of officers, 188 by Fellows and 656 by Members. The officers elected to serve in 1960 are:

President: Joseph Murdoch, University of California at Los Angeles, California.
Vice-President: E. F. Osborn, The Pennsylvania State University, University Park, Pennsylvania.
Secretary: George Switzer, U. S. National Museum, Washington 25, D. C.
Treasurer: Marjorie Hooker, U. S. Geological Survey, Washington 25, D. C.
Councilors: (1960–1962) Julian R. Goldsmith, University of Chicago, Chicago, Illinois
Horace Winchell, Yale University, New Haven, Connecticut

According to the provisions of the Constitution, the following have been elected to fellowship:

Robert Marshall Douglass, Los Alamos Scientific Laboratory, Los Alamos, New Mexico.
Richard Clarkson Erd, U. S. Geological Survey, California.
Gerald Manfred Friedman, Pan American Petroleum Company, Tulsa, Oklahoma.
Erwin Emil Hellner, University of Chicago, Chicago, Illinois.
Vernon James Hurst, Department of Mines, Atlanta, Georgia.
Edward Charles Jonas, The University of Texas, Austin, Texas.
Solomon Kaiman, Mines Branch, Ottawa, Canada.
Donald Richard Lewis, Shell Development Company, Houston, Texas.
Katharine Selden Kniskern Mather, U. S. Army Engineer Waterways Experiment Station, Jackson, Mississippi.

Peter Misch, University of Washington, Seattle, Washington.
Peter Aubrey Sabine, Geological Survey of Great Britain, London, England.
Leonard B. Sand, University of Utah, Salt Lake City, Utah.
Robert Leland Smith, U. S. Geological Survey, Washington, D. C.
Roland Bliss Snow, U. S. Steel Corporation, Monroeville, Pennsylvania.
W. Ehrenreich Tröger, University of Freiburg, Germany.
Tómas Tryggvason, University Research Institute, Reykjavik, Iceland.
Alexis von Volborth, University of Nevada, Reno, Nevada.
Marc Bernard Vuagnat, University of Lausanne, Switzerland.
Donald Edward White, U. S. Geological Survey, Washington, D. C.
Harold Douglas Wright, Pennsylvania State University, University Park, Pennsylvania.
E-an Zen, University of North Carolina, Chapel Hill, North Carolina.

MEMBERSHIP STATISTICS

November 1, 1959

	1958	1959	Gain	Loss
Correspondents	3	0	0	3
Honorary Fellows	0	3	4	1
Fellows	360	376	21	5
Members	1,185	1,501	416	100
Subscribers	1,195	1,339	227	83

The above figures show a net gain of 16 fellows, 316 members and 144 subscribers. Considering the four groups together, there is a gain of 476 giving a total of 3219.

The accompanying Membership Chart shows graphically the growth of the Society. With the exception of the war years 1940–'42 each year has seen an increase over the preceding year in members, fellows and subscribers to the *American Mineralogist* with 1959 showing the greatest increase in a single year.

During 1959, the Society lost through death six fellows: Charles R. Fettke, Pittsburgh, Pennsylvania; Peggy-Kay Hamilton, Bronxville, New York; Charles Mauguin, Thiais, France; James E. Maynard, Syracuse, New York; L. J. Spencer, London, England; Samuel Zerfoss, Washington, D. C.

Respectfully submitted,
C. S. Hurlbut, Jr., *Secretary*

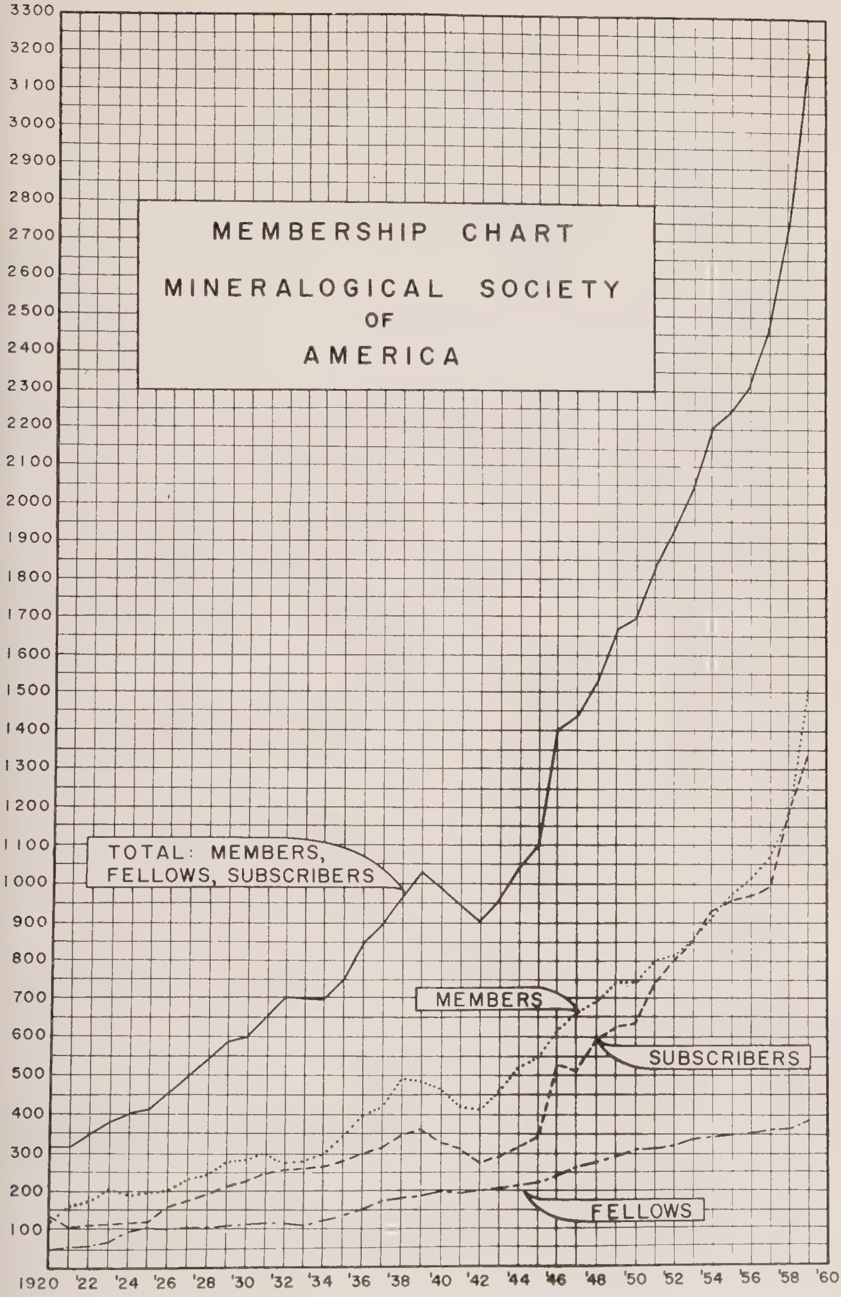
REPORT OF THE TREASURER FOR 1959

To the Council of the Mineralogical Society of America:

Your treasurer submits herewith her report for the fiscal year beginning August 1, 1958, and ending July 31, 1959.

Summary Statement

Cash on hand, August 1, 1958		$ 1,599.14
Total receipts	$ 40,855.23	
Total disbursements	35,450.46	
Net income (Receipts less disbursements)	$ 5,404.77	$ 5,404.77
Total income		$ 7,003.91


MEMBERSHIP CHART
MINERALOGICAL SOCIETY OF AMERICA
TOTAL: MEMBERS, FELLOWS, SUBSCRIBERS
MEMBERS
SUBSCRIBERS
FELLOWS
3300
3200
3100
3000
2900
2800
2700
2600
2500
2400
2300
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
1920
'22
'24
'26
'28
'30
'32
'34
'36
'38
'40
'42
'44
'46
'48
'50
'52
'54
'56
'58
'60

Cash on hand, July 31, 1959		
Riggs National Bank	$ 1,502.67	
Merrill Lynch, Pierce, Fenner and Smith	428.48	$ 1,931.15
Transfer to capital		5,072.76
Total		$ 7,003.91
Assets, July 31, 1959		
Cash	$ 1,931.15	
Capital (Endowment Fund securities and cash)	148,902.50	
Total		$150,833.65

Financially, the society has completed a year marked by progress in several respects. The figures on income, expenditures, and balances are shown in the tables. The explanatory comments that follow are to provide background information and point out some percentage relationships and trends.

Income and Expense

The effective income of the society this year can be grouped in three categories and was obtained from these sources directly or from activity concerned with them.

Dues	18.2%
Publication program	56.2
Endowment fund	25.6

Dues furnished 18.2 per cent of income, and while no comparison can be made with previous years when income from dues and subscriptions was not reported separately, it is considerably more than that calculated on the basis of membership figures. The receipt of $1100 more than a normally expectable $6300 is the direct result of a net gain of 275 members between January and July 1959. Income from various sources in connection with publications amounts to 56.2 per cent. The lower income from sale of reprints, compared to the previous year, is due to the difference in time of invoicing and receipt of payments in relation to fiscal year closing. The income from the sale of indexes is more than twice that received last year. Income recorded under the endowment fund made up the remaining 25.6 per cent and will be discussed separately.

Expenditures can be grouped as follows:

Officers' honoraria and office expense	12.1%
Publication program	85.3
Support of other organizations and miscellaneous	2.6

The largest portion of the publication expense is naturally that of printing and distributing the *American Mineralogist*, amounting this year to $23,341.19 or about 77 per cent. As a result of the increased cost of our journal, the grant of $8000 from the Geological Society of America toward its publication now defrays one-third rather than one-half of the cost. The expenditure for postage shows a large increase over last year (44 per cent), resulting from the increases in postal rates, sales of indexes, and membership.

Endowment Fund

Colonel Roebling's original gift of $45,000 in 1926 formed the nucleus of the endowment fund which has since grown by continual investment of money from the society's operating fund. In November 1958 the Fifty Plus Committee was initiated privately for the explicit purpose of increasing the endowment fund by asking a minimum pledge of $50.00 from individual members, to be contributed over a five-year period. At the close of the fiscal year, $11,600 had been pledged and $3719 of this had been received. Voluntary contributions during the same period supplied an additional $240.50.

The total book value of all securities in the fund at the end of the fiscal year was $118,956.68. Of this amount, 12.9 per cent was in bonds, 28.7 per cent in preferred stocks, and 58.4 per cent in common stocks. The approximate market value, computed on July 1, 1959, was $147,944.00. The higher figure of the market value is accounted for entirely by the common stocks, and the good position of the society in this respect can be attributed to the recommendations of the Finance Committee in this direction and the policy of the preceding treasurer to increase the holdings in common stocks. Dividends and interest accruing from the investments amounted to $6518.42 and accounted for 16 per cent of total income. The yield for the year at book value was 5.48 per cent; at market value, 4.41 per cent. From securities now held an income of $6850 is estimated for 1959–1960.

To simplify the details of managing the portfolio which now numbers thirty-two securities, a custodian account was opened with Merrill Lynch, Pierce, Fenner, and Smith, Inc. of September 5, 1958.

Mineralogical Abstracts

Beginning with the issue of volume 14, number 1, March 1959, our society assumed joint responsibility with the Mineralogical Society of Great Britain and Ireland for the publication of Mineralogical Abstracts separately from the Mineralogical Magazine. As shown in the statement, the society has made an advance of funds toward initial publication expense. It remains to be seen whether the support of Mineralogical Abstracts will be a recurrent annual expense or whether the venture will become self-supporting in the near future. During the year, the treasurer also acted as agent for the collection and transfer of subscription payments from our members.

I am most fortunate in having the complete cooperation of our Finance Committee and Merrill Lynch, custodian of our securities, and it is a pleasure for me to record here my thanks to Edward P. Henderson, Samuel S. Goldich, and Arthur Montgomery of the committee, and to Stanly Carr of Merrill Lynch, for their competent advice and assistance at all times.

RECEIPTS

Dues		
Fellows	$1,485.40	
Members	5,929.91	$ 7,415.31
Publications		
Geological Society of America grant	8,000.00	
Subscriptions to American Mineralogist	7,609.29	
Sale of reprints	2,698.97	
Sale of back numbers	2,776.21	
Sale of Index, vols. 31–40	707.40	
vols. 21–30	261.00	
vols. 1–20	138.60	
Advertising	746.06	
Miscellaneous	20.02	22,957.55

Endowment Fund		
Dividends and interest	6,518.42	
Fifty-Plus Committee	3,719.00	
Contributions	240.50	10,477.92
Exchange credits and collections	4.45	4.45
Total receipts		$40,855.23

DISBURSEMENTS

Honoraria		
Secretary (August 1, 1957–July 31, 1958)	$ 250.00	
Treasurer (August 1, 1957–November 15, 1958)	322.91	
Editor (July 1, 1958–March 31, 1959)	562.50	$ 1,135.41
Office Expense		
Clerical assistance (April 1958–July 1959)	1,434.20	
Printing and stationery	979.80	
Supplies	70.99	
Certificates of Incorporation (3)	10.50	
Officers' travel	198.26	
Committee expense	7.91	
Telephone and telegrams	5.95	
Postage	438.64	3,146.25
Publications		
American Mineralogist (July 1958–June 1959)	23,341.19	
Reprints	3,358.38	
Program and abstracts	400.00	
Microtext of out-of-print issues	590.86	
Purchase of out-of-print issues	88.00	
Postage and express	358.67	
National Science Foundation refund on index sales (May 1958–April 1959)	693.90	
Mineralogical Abstracts (publication advance)	1,405.38	30,236.38
International Mineralogical Association	120.00	
American Geological Institute	594.00	714.00
Roebling Medal	150.63	
MSA Award certificate	7.50	158.13
Refunds	42.29	
Checks returned	18.00	60.29
Total disbursements		$35,450.46

ENDOWMENT FUND

Amount	Security	Cost	Value[1]	Income[2]
	Bonds			
6M	Atlantic Coast Line, 4½	$ 5,257.50	$ 6,000	$ 270.00
5M	New York Central, 5	4,300.00	3,600	250.00
5M	Southern Railway, 5	5,743.75	4,800	250.00
		$ 15,301.25	$ 14,400	$ 770.00

	Preferred stocks			
500	Union Pacific, 4	4,570.25	4,000	200.00
200	Southern California Edison, 4.88	5,250.00	5,000	244.00
150	Fairbanks Whitney	4,487.50	3,450	120.00
60	Jones and Laughlin, A5	4,987.50	5,940	300.00
55	United States Steel, 7	6,946.20	7,865	385.00
50	Virginia Electric Power, 5	5,942.50	5,150	250.00
40	Potomac Electric Power, 2.44	2,000.00	1,960	97.60
	Consolidated Edison, 5	—	—	37.50[3]
		$ 34,183.95	$ 33,365	$1,634.10
	Common stocks			
350	Potomac Electric Power	5,066.73	9,100	415.20
261	Kroger Company	2,034.03	7,569	219.69
249	American Telephone and Telegraph	11,255.98	19,920	765.68
230	Columbia Gas	3,764.56	5,060	215.00
200	Greyhound	2,300.00	4,600	200.00
180	Diana Stores	2,800.30	3,420	100.00[4]
175	Standard Oil of New Jersey	1,761.65	9,100	393.75
150	Consolidated Denison	3,096.00	2,250	—
100	Buckeye Steel Castings	3,800.00	2,800	150.00
100	Columbus and Southern Ohio	2,087.50	3,400	160.00
100	Pittsburgh and West Virginia	2,787.50	2,100	—
100	Plymouth Cordage	4,750.00	4,500	260.00
100	Southern Natural Gas	3,600.00	3,900	200.00
60	United Fruit	3,067.50	2,040	135.00
50	Chesapeake and Ohio Railway	2,368.75	3,600	200.00
50	Phelps Dodge	1,975.00	3,050	150.00
50	Sinclair Oil	2,968.75	3,050	150.00
50	Public Service Electric and Gas	1,586.40	1,950	90.00
50	Spencer Kellogg	1,258.33	900	40.00
50	Boston Insurance	1,500.00	1,650	90.00
50	Chase Manhattan Bank	3,231.25	3,100	30.00[5]
30	U. S. Playing Card	2,411.25	3,120	150.00
		$ 69,471.48	$100,179	$4,114.32
	Total	$118,956.68	$147,944	$6,518.42
	Cash balance, Endowment Fund account, Riggs National Bank	958.50		

[1] Approximate market value, July 1, 1959.

[2] Fiscal year ending July 31, 1959, except where noted.

[3] Income for 3 quarters. Sold in March 1959.

[4] Income on 100 shares. Additional shares purchased June 1959.

[5] Income for one quarter only. Purchased in March 1959.

Respectfully submitted,
Marjorie Hooker, *Treasurer*

REPORT OF THE AUDITING COMMITTEE

To the President of the Mineralogical Society of America:

The Auditing Committee has examined and verified the accounts of the Treasurer of the Mineralogical Society of America for the fiscal year beginning August 1, 1958, and ending July 31, 1959. The securities listed in the Treasurer's report, with all future coupons on the coupon bonds attached, are in the safety deposit box at the 17th and G Street Branch of the Riggs National Bank in Washington, D. C.

Respectfully submitted,
G. Kullerud
E. W. Roedder
M. E. Mrose, *Chairman*

REPORT OF THE EDITOR FOR 1959

The 1959 volume of *The American Mineralogist* contained 1344+vii pages. As shown in the summary, 1237 of these were devoted to articles. This figure represents an increase of nearly 200 pages over Volume 43. There were 75 major and 67 shorter articles, giving a total of 142. The authors represented 26 U. S. educational institutions, 10 government laboratories, 11 industrial laboratories, 15 foreign educational institutions and 8 foreign laboratories, giving a total of 70 institutions. This compares with 37 in 1957 and 54 in 1958 It is evident that we have an international journal.

Seven new minerals were described in detail for the first time in Volume 44. There were 7 book reviews. The very important section on New Mineral Names, so ably conducted by Dr. M. Fleischer, contained 70 entries. The value of these abstracts is greatly enhanced by his critical comments.

As in former years, the Geological Society of America has continued its liberal financial support toward the cost of printing the journal, and we are greateful for this assistance.

With approximately 200 more pages devoted to articles in Volume 44, the backlog of manuscripts has been somewhat reduced. At present, most of the shorter articles appearing under Notes are printed within four months of receipt. The waiting period for major articles has varied from seven to ten months, with the majority appearing within eight months. We hope to be able to get this period down to six months.

The associate editors have been called upon to read many manuscripts, and to make recommendations to the editor and to the authors. Our thanks are extended to these individuals, Robert Garrells, Joseph Murdoch, D. Jerome Fisher, George T. Faust, George W. Brindley and Adolf Pabst. The editor has also called on others to help in evaluating manuscripts, including C. L. Christ, R. M. Denning, H. T. Evans, E. Ingerson, P. F. Kerr, B. Mason, R. S. Mitchell, J. V. Smith, O. F. Tuttle and H. Winchell.

The editor likewise wishes to express his appreciation for the splendid cooperation he receives from the publisher, The George Banta Company, Inc., and for the excellent appearance of the journal.

A summary of the contents of Vol. 44 is given in the following table.

Distribution of Subject Matter in Volume 44

Subjects	Number	Pages		% of Total
Leading articles	75	1,040		78.2
Short articles	67	197		14.8
			1,237	93.0
Memorials	5	19		
Awards	2	9		
Proceedings of the Society	1	17		
			45	3.45
Book Reviews	7	9		
New Mineral Names	70	29		
Notes	12	9		
			47	3.55
Total			1,329	100.00
Index, Title Page, Table of Contents			22	
Grand Total			1,351	

Respectfully submitted,
Lewis S. Ramsdell, *Editor*

LIST OF FORMER OFFICERS AND MEETING PLACES

By recommendation of the Council, a complete list of past officers is printed in the proceedings of the annual meeting of the Society:

Honorary Presidents

Edward S. Dana	1925–1935
Charles Palache	1949–1954
Edward H. Kraus	1955–

Presidents		Vice-Presidents	
1920	Edward H. Kraus	1920	Thomas L. Walker
1921	Charles Palache	1921	Waldemar T. Schaller
1922	Thomas L. Walker	1922	Frederick A. Canfield
1923	Edgar T. Wherry	1923	George F. Kunz
1924	Henry S. Washington	1924	Washington A. Roebling
1925	Arthur S. Eakle	1925	Herbert P. Whitlock
1926	Waldemar T. Schaller	1926	George Vaux, Jr.
1927	Austin F. Rogers	1927	George L. English
1928	Esper S. Larsen	1928	Lazard Cahn
1929	Arthur L. Parsons	1929	Edward Wigglesworth
1930	Herbert E. Merwin	1930	John E. Wolff
1931	Alexander H. Phillips	1931	William F. Foshag
1932	Alexander N. Winchell	1932	Joseph L. Gillson
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1934	John W. Wolff	1934	William A. Tarr
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1936	William S. Bayley	1936	Harold L. Alling
1937	Norman L. Bowen	1937	H. V. Ellsworth
1938	Ellis Thomson	1938	Kenneth K. Landes
1939	Max N. Short	1939	Burnham S. Colburn
1940	William F. Foshag	1940	Ian Campbell
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1944	R. C. Emmons	1944	Harry Berman
1945	Kenneth K. Landes	1945	George Tunell
1946	Paul F. Kerr	1946	S. B. Hendricks
1947	M. J. Buerger	1947	Carl Tolman
1948	M. A. Peacock	1948	Adolf Pabst
1949	John W. Gruner	1949	J. D. H. Donnay
1950	George Tunell	1950	Ralph E. Grim
1951	A. Pabst	1951	Michael Fleischer
1952	Michael Fleischer	1952	J. D. H. Donnay
1953	J. D. H. Donnay	1953	Sterling B. Hendricks
1954	Sterling B. Hendricks	1954	Harry H. Hess
1955	Harry H. Hess	1955	Clifford Frondel
1956	Clifford Frondel	1956	D. Jerome Fisher
1957	D. Jerome Fisher	1957	George E. Goodspeed
1958	George E. Goodspeed	1958	Ralph E. Grim
1959	Ralph E. Grim	1959	Joseph Murdoch

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1933–1934	Albert P. Peck
1934–1944	Paul F. Kerry
1944–1959	C. S. Hurlbut, Jr.
1959–	George Switzer

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1924–1929	Alexander H. Phillips
1929–1930	Albert B. Peck
1930–1940	Waldemar T. Schaller
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1923 Alexander H. Phillips, Austin F. Rogers, Thomas L. Watson, Esper S. Larsen.
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1933 Paul F. Kerr, William S. Bayley, William J. McCaughey, Kenneth K. Landes.
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1935 William J. McCaughey, Kenneth K. Landes, E. P. Henderson, J. F. Schairer.
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1937 E. P. Henderson, J. F. Schairer, Arthur F. Buddington, Arthur P. Honess.
1938 J. F. Schairer, Arthur F. Buddington, Arthur P. Honess, R. C. Emmons.
1939 Arthur F. Buddington, Arthur P. Honess, R. C. Emmons, Carl Tolman.
1940 Arthur P. Honess, R. C. Emmons, Carl Tolman, D. Jerome Fisher.
1941 R. C. Emmons, Carl Tolman, D. Jerome Fisher, Martin A. Peacock.
1942 Carl Tolman, D. Jerome Fisher, Martin A. Peacock, Adolf Pabst.
1943 D. Jerome Fisher, Martin A. Peacock, Adolf Pabst, C. S. Hurlbut, Jr.
1944 Martin A. Peacock, Adolf Pabst, Michael Fleischer, S. J. Shand.
1945 Adolf Pabst, Michael Fleischer, S. J. Shand, R. E. Grim.
1946 Michael Fleischer, S. J. Shand, R. E. Grim, Joseph Murdoch.
1947 S. J. Shand, R. E. Grim, Joseph Murdoch, H. H. Hess.
1948 R. E. Grim, Joseph Murdoch, H. H. Hess, Clifford Frondel.
1949 Joseph Murdoch, H. H. Hess, Clifford Frondel, Lewis S. Ramsdell.
1950 H. H. Hess, Clifford Frondel, Lewis S. Ramsdell, E. F. Osborn.
1951 Clifford Frondel, Lewis S. Ramsdell, E. F. Osborn, George T. Faust.
1952 Lewis S. Ramsdell, E. F. Olson, George T. Faust, Victor T. Allen.
1953 E. F. Osborn, George T. Faust, Victor T. Allen, C. Osborn Hutton.
1954 George T. Faust, Victor T. Allen, C. Osborne Hutton, Felix Chayes.
1955 Victor T. Allen, C. Osborne Hutton, Felix Chayes, L. G. Berry.

1956 C. Osborne Hutton, Felix Chayes, L. G. Berry, C. B. Slawson, A. O. Woodford.
1957 Felix Chayes, L. G. Berry, C. B. Slawson, A. O. Woodford, S. S. Goldich.
1958 L. G. Berry, C. B. Slawson, A. O. Woodford, S. S. Goldich, B. H. Mason, R. H. Jahns C. Milton.
1959 S. S. Goldich, B. H. Mason, R. H. Jahns, C. Milton, W. R. Foster, E. W. Nuffield.

Annual Meeting Places

1920 Chicago, Illinois
1921 Amherst, Massachusetts
1922 Ann Arbor, Michigan
1923 Washington, D. C.
1924 Ithaca, New York
1925 New Haven, Connecticut
1926 Madison, Wisconsin
1927 Cleveland, Ohio
1928 New York, N. Y.
1929 Washington, D. C.
1930 Toronto, Canada
1931 Tulsa, Oklahoma
1932 Cambridge, Massachusetts
1933 Chicago, Illinois
1934 Rochester, New York
1935 New York, N. Y.
1936 Cincinnati, Ohio
1937 Washington, D. C.
1938 New York, N. Y.
1939 Minneapolis, Minnesota
1940 Austin, Texas
1941 Boston, Massachusetts
1942 No meeting held
1943 No meeting held
1944 No meeting held
1945 Pittsburgh, Pennsylvania
1946 Chicago, Illinois
1947 Ottawa, Canada
1948 New York, N. Y.
1949 El Paso, Texas
1950 Washington, D. C.
1951 Detroit, Michigan
1952 Boston, Massachusetts
1953 Toronto, Canada
1954 Los Angeles, California
1955 New Orleans, Louisiana
1956 Minneapolis, Minnesota
1957 Atlantic City, New Jersey
1958 St. Louis, Missouri
1959 Pittsburgh, Pennsylvania

Recipients of the Roebling Medal

1937 Charles Palache
1938 Waldemar T. Schaller
1940 Leonard James Spencer
1941 Esper S. Larsen, Jr.
1945 Edward H. Kraus
1946 Clarence S. Ross
1947 Paul Niggli
1948 William Lawrence Bragg
1949 Herbert E. Merwin
1950 Norman L. Bowen
1952 Fred E. Wright
1953 William F. Foshag
1954 Cecil Edgar Tilley
1955 Alexander N. Winchell
1956 Arthur F. Buddington
1957 Walter F. Hunt
1958 Martin J. Buerger
1959 Felix Machatschki

Recipients of the Mineralogical Society of America Award

1951 Orville F. Tuttle
1952 Frederick H. Stewart
1953 L. H. Ahrens
1954 Hatten S. Yoder, Jr.
1955 Julian R. Goldsmith
1956 George C. Kennedy
1957 Rustum Roy
1958 Charles E. Weaver
1959 Harry F. W. Taylor

1959 ANNUAL MEETING AT PITTSBURGH, PENNSYLVANIA

The meetings of the following societies were held on November 2–4, 9959.

The Geological Society of America—72nd
The Paleontological Society—51st
The Mineralogical Society of America—40th
Society of Economic Geologists—40th
The Geochemical Society—4th

The complete program listed titles and abstracts of 323 papers. Of these 145 seemed to be of special interest to members of The Mineralogical Society of America. Authors and titles of these papers are listed below. Since by Council action the abstracts will not be reprinted in *The American Mineralogist*, references are given to the *Bulletin of the Geological Society of America*, Vol. 70, Number 12, Part 2, December 1959, pp. 1559–1706, where the abstracts of all the papers of the various societies are reprinted, arranged alphabetically by authors.

PAPERS OF SPECIAL MINERALOGICAL INTEREST

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VICTOR T. ALLEN AND WM. D. JOHNS: *Clay-mineral environments in New England and eastern Canada.* Page 1560.

SHIGEO ARAMAKI AND RUSTUM ROY: *Detailed x-ray data on mullites and two new anhydrous alumino-silicates AS(H)-II and high-temperature sillimanite.* Page 1562.

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NOTES AND NEWS

VONSENITE FROM ST. LAWRENCE COUNTY, NORTHWEST ADIRONDACKS, NEW YORK*

B. F. LEONARD, *U. S. Geological Survey, Denver, Colorado* AND
ANGELINA C. VLISIDIS, *U. S. Geological Survey,*
Washington, D. C.

The opaque mineral vonsenite, a high-temperature ferrous ferric borate of the ludwigite-vonsenite series, occurs in skarn in the pyrometasomatic magnetite deposit at Jayville, St. Lawrence County, New York. Chemical and microscopic data suggest that vonsenite is a major constituent of at least part of the chief magnetite-bearing zone at Jayville. A mineral of identical optical properties is present at the Clifton mine, near Degrasse, St. Lawrence County, New York. The geology of the district and its ore deposits is described in reports in preparation by Buddington and Leonard. Their account of the Jayville deposit is based on unpublished geologic mapping by Tyler and Wilcox [1942].

Vonsenite was found at Jayville by Leonard in 1947 in drill core from hole J 2 of the Jones and Laughlin Steel Corporation. The mineral was identified in 1951 by the *x*-ray work of J. M. Axelrod and spectrographic analysis by Janet D. Fletcher, both of the U. S. Geological Survey.

In hand specimen, the vonsenite looks the same as the fine-grained magnetite with which it occurs. The vonsenite forms black, granular aggregates with metallic (locally adamantine) luster. Powdered vonsenite will move when an alnico magnet is run along a glass tube containing the pure mineral.

Vonsenite is completely opaque in thin section. If the thin section is viewed in incident light, one sees that vonsenite is darker than the associated magnetite. If one examines polished specimens of ore with the naked eye, one sees that vonsenite is bluish relative to magnetite, and its reflectivity or "brightness" relative to magnetite is about the same as the reflectivity of magnetite relative to crystalline hematite.

In polished sections examined with the reflecting microscope, vonsenite shows the following properties: *Habit*—generally aggregates of polygonal grains; locally lath-, wedge-, and diamond-shaped crystals, commonly with rounded corners. Apparently contemporaneous with, or slightly younger than magnetite; cut by veinlets of hematite, and locally replaced by ragged blades of hematite. *Color*—very strongly pleochroic from light gray or light brown to light blue-gray. *Reflectivity*—maximum (light brown direction) about 13–16 per cent, minimum (light blue-gray direction) about 10–12 per cent for green light. (These are preliminary measurements, subject to correction.) *Anisotropism*—very strong or extreme, from black to fiery orange. Extinction parallel to long edges of laths, but symmetrical in wedges and diamonds. No internal reflection. *Hardness*—scratching hardness F or greater, or about 6 on Mohs' scale. Polishing hardness less than that of hematite, and about equal to that of magnetite. *Magnetism*—pure mineral powder readily attracted to magnetized sewing needle. *Etch tests*—HCl positive; aqua regia weakly positive; HNO_3, $FeCl_3$, KOH, $HgCl_2$, H_2O_2 negative. Without *x*-ray, chemical, or refined optical data, the vonsenite would easily be mistaken for ilvaite. (Cf. Ramdohr, 1955, p. 811–812.)

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* Publication authorized by the Director U. S. Geological Survey.

A chemical analysis of 1.9 grams of nearly pure vonsenite, separated by W. T. Schaller, is given in Table 1. (The sample is field no. J 2.21, hole J 2, depth 299 feet; laboratory sample no. 144483; laboratory report IWC-473.)

Table 1. Chemical Analysis of Vonsenite

	Wt. per cent	Wt. per cent (recalc.)
FeO	53.06	54.04
MnO	0.27[a]	0.28
MgO	0.55[b]	0.56
Fe_2O_3	29.59	30.14
Al_2O_3	1.45	1.48
TiO_2	0.13	0.13
SnO_2	Present[c]	Present
B_2O_3	13.13	13.37
CaO	None	100.00
Acid insoluble	1.80	
	99.98	

Angelina C. Vlisidis, analyst, 1955

G. of analyzed sample = 4.75 }
G. corrected for acid insoluble = 4.77 } W. T. Schaller

[a] Amount of MnO recovered from the Mg precipitate. Total amount not determined.

[b] Corrected for the contained MnO (0.27 per cent).

[c] Not determined gravimetrically. Spectrographic determination (see below) indicates a small quantity, about 0.x per cent.

The recalculated analysis yields the oxide ratios 4.01:1.07:1.00, close to the ideal $4FeO \cdot Fe_2O_3 \cdot B_2O_3$. The composition may be expressed as $4[(Fe''_{1.92}Mn_{.01}Mg_{.04})(Fe'''_{.96}Al_{.07}Ti_{.004})B_{.98}O_5]$, based on the structural formula of daSilva, Clark, and Christ (1955).

Spectrographic analysis, made in 1951 by Miss Fletcher, shows (report IWS-156):

	Per cent
Fe, B	>5
Mg, Si, Al	1–5
Sn, Pb, Zn, Mn, Ti	0.x
Cu, Cr, Zr, Ba, Ca	0.0x
Ag, Co, Ni	0.00x

Looked for but not found: As, Bi, Be, Au, Pt, Mo, W, Ge, Sb, Cd, Tl, Ga, V, Y, La, Th, Cb, Ta, U, Sr, Na, P.

In chemical composition, the Jayville vonsenite closely approaches the $Fe_2'' Fe''' BO_5$ end-member of the ludwigite-vonsenite series. We use the term vonsenite on the advice of Dr. Schaller. The statement in Dana VII, **2** (Palache, Berman, and Frondel, 1951, p. 321; private communication from W. T. Schaller) that ludwigite and paigeite are isostructural with a complete series extending between them is incorrect, as shown by later studies and better *x*-ray diffraction patterns (Schaller, W. T., 1955, personal communication to Leonard). The statement should read: ludwigite and vonsenite are isostructural, and a complete series extends between them by mutual substitution of Mg and Fe″. The data listed for no. 24.1.1.2 in Dana VII, **2** (p. 322) refer to ludwigite (Mg end-member) and to vonsenite (Fe″ end-member), not to paigeite.

Table 2. *X*-Ray Powder Diffraction Data for Jayville Vonsenite
(FeKα radiation and Mn filter)

d	*I*	*d*	*I*
7.52	w	2.07	ms
6.16	w	2.01	w
5.17	s	1.940	ms
3.03	w	1.767	w
2.85	mw	1.602	mw
2.58	vs	1.539	ms
2.38	ms	1.504	mw
2.18	ms	1.404	mw

The measurements of these stronger lines were made by Fred A. Hildebrand, U. S. Geological Survey, on a film taken by him on a part of the analyzed sample. They are to be regarded only as cursory measurements.

Vonsenite from Riverside, California, was originally described by Eakle (1920). "Paigeite" (vonsenite) from the Kamaishi mine, Japan, was described by Watanabe and Ito (1954). Federico (1957) showed that the breislakite of Vesuvius and Capo di Bove, Italy, is vonsenite. Later (Federico, 1958), she studied the alteration of breislakite and synthetic vonsenite. Vakar, Knipovich, and Shafranovskii (1934) described as ludwigite material whose $(Fe''+Mn)/Mg$ ratio relative to 5 oxygens is about 0.99/0.84. Pure vonsenite has been synthesized by Andrieux and Weiss (1944), Chevallier, Mathieu, and Girard (1948), Bertaut, Bochirol, and Blum (1950), and Federico (1958).

We wish to thank the Jones and Laughlin Steel Corporation, the Benson Iron Ore Corporation, and our colleagues in the Geological Survey for their cooperation in this study. A complete account of the Jayville vonsenite will appear in another journal.

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PSEUDOMORPHES AFTER DATOLITE, PREHNITE AND APOPHYLLITE FROM EAST GRANBY, CONNECTICUT

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Introduction

In the spring of 1958, during the annual spring field trip of the Boston University Geology Department, one of the stops of the group was at the East Granby, Connecticut, trap rock quarry of Materials Service, Inc. During the quarry examination, the group was met by Mr. Stephen Ridel, one of the employees in the quarry, who makes a practice of collecting a completely representative suite of minerals in the quarry. Following the exploration of the quarry, the group was invited to visit Mr. Ridel's personal collection at his home. Through the years, the quarry has produced some outstanding specimens of the usual trap rock minerals Mr. Ridel presented two specimens for identification which could not be immediately recognized, and specimens were kindly made available for study in the Boston University Mineralogy Laboratories. Specimens studied are shown in Fig. 1*a*, 1*b*.

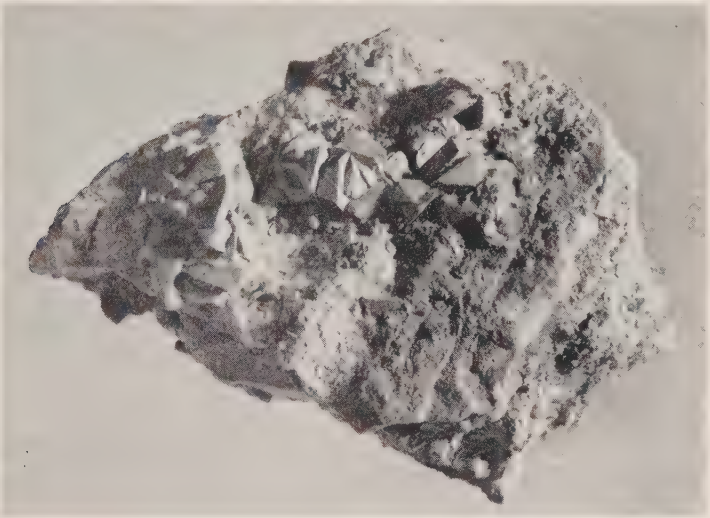

Fig. 1*a*. Chlorite pseudomorphs after datolite on veined pocket filling. Specimen 6 inches wide.

DESCRIPTION OF SPECIMENS

The trap rock quarry at East Granby is of the typical Triassic basaltic variety. At the time the authors were examining the quarry, there were but few evidences of cavities or extensive calcite or quartz veination. Pockets with mineralization, according to Mr. Ridel, are occasionally discovered, and it was from one of these that the materials herein described were removed.

The outstanding feature of the material considered herewith is the abundance of chlorite. The materials show signs of successive shearing and vein filling with calcite as the predominant vein filler with minor amounts of clear and slightly smoky quartz crystals lining some of the shear openings. Maximum length of these quartz crystals in the specimens at hand is about 4 mm. Rosettes of both white and green prehnite, up to 1 cm. across, are common in the material, Fig. 1*b*.

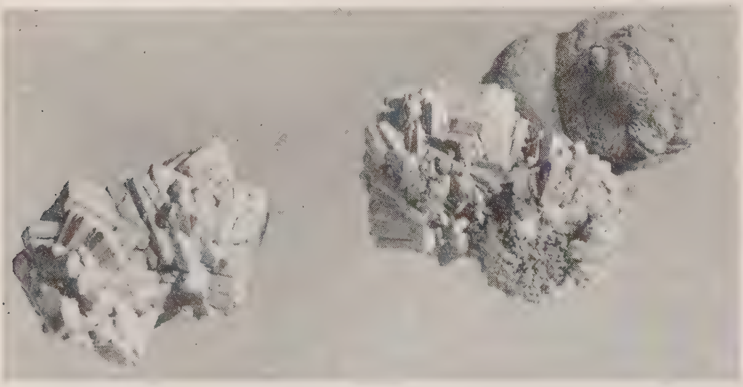

FIG. 1*b*. Chlorite pseudomorphs after apophyllite and prehnite on veined pocket filling.

ALTERATION

The material of particular interest in this paper is a series of specimens with strange satiny luster and green coloration, typical of chlorite pseudomorphs after adularia, and a tentative identification of the crystals as adularia was made. The crystals were studied under the polarizing microscope, which quickly disclosed their pseudomorphic character. At least two substances are present in the crystals, but the grain size of individual units is too small to permit accurate determination by optical means. The optical data were adequate, however, to leave no doubt that the replacing material is chlorite.

The precise sequence of alteration in the matrix of the altering medium cannot be certainly determined from the data at hand. The most important change has been the extensive addition of Fe^{+2} in the process. Since zeolites and associated minerals are low density, water-bearing minerals, rich in sialic constituents, it appears that they are formed under low pressures and temperatures, probably due to the migration of meteoric water through the rocks. This water dissolves out the sialic constituents which are in turn precipitated in the gas cavities. A subsequent and deeper burial of the lavas with a rise in temperature and pressure would subject the zeolites and associated minerals to unstable conditions. Under these conditions a 2-phase migration of ions would take place with the sialic ions migrating upward and iron and magnesium ions migrating downward. The pseudomorphic alteration is visualized as being essentially due to this substitution of iron and magnesium for calcium, sodium and potassium.

MORPHOLOGICAL CRYSTALLOGRAPHY

On the basis of the crystallographic measurements, it was decided that two of the substances had originally been datolite and apophyllite. The pseudomorphs after prehnite were determined from the usual growth habit of prehnite. A comparison of the measured angular values with those listed in the Dana 1892 *System of Mineralogy* is given below.

Comparison of Interfacial Angles

	Datolite (*Dana*)		*Datolite Pseudomorphs*
$n \wedge n'$	$111 \wedge 1\bar{1}1$	59°4½′	60°
*$(n \wedge n'$	$111 \wedge 111)$		
$a \wedge n$	$100 \wedge 111$	38°55′	40°
$(c \wedge n$	$001 \wedge 111)$		
$a \wedge m$	$100 \wedge 110$	32°23½′	36°
$(c \wedge M$	$001 \wedge 011)$		
$\epsilon \wedge \epsilon'$	$\bar{1}12 \wedge \bar{1}\bar{1}2$	48°19½′	48°
$(\epsilon \wedge \epsilon'$	$\bar{2}11 \wedge \bar{2}\bar{1}1)$		

* Letters and indices in parentheses refer to orientation with $c < a$.

The optical goniometric measurements of the datolite gave very diffuse signals, and readings were to the nearest degree. These measurements were then plotted on the Wulff net, and the interfacial angles were measured thereon. Four crystals were measured in all, and there was a complete consistency of results. The data given above were those from one crystal. Figure 2 represents this crystal in the orientation $c < a$ but with Dauber's (Dana) unit.

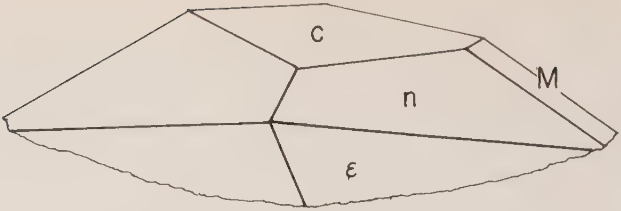

FIG. 2. Typical chlorite pseudomorph after datolite crystal.

Comparison of Interfacial Angles

	Apophyllite (after Dana)		*Apophyllite Pseudomorphs*
$p\wedge p'$	$111\wedge 111$ *$(011\wedge 101)$	76°0′	73°
$c\wedge p$	$001\wedge 111$ $(001\wedge 011)$	60°30′	60°

* Indices in parenthesis refer to structural orientation which is used in Fig. 3.

The measurements of the apophyllite were done by the use of the contact goniometer and were completely adequate to demonstrate the identity of the pseudomorphs. Figure 3 shows the typical habit of the individual apophyllite crystals which actually occur in clusters. It should be noted that the (111) of Dana is actually (101) of the structural cell.

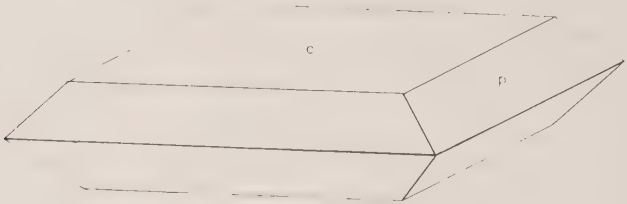

FIG. 3. Typical chlorite pseudomorph after apophyllite crystal.

The variation in density of the pseudomorphs as compared with the original crystals gives some suggestion as to the degree of replacement by chlorite. It was impossible to get a precision determination of the chlorite composition, but a specific gravity of roughly 2.8 seems reasonable in terms of the final densities. A comparison of the altered and unaltered crystals appears below.

Comparison of Specific Gravities

Datolite (after Dana) 3.0	*Datolite Pseudomorphs* 2.885
Prehnite (after Dana) 2.8–2.95	*Prehnite Pseudomorphs* 2.89
Apophyllite (after Dana) 2.3–2.4	*Apophyllite Pseudomorphs* 2.51

It will be noted that whereas the datolite pseudomorphs have a lower specific gravity than the original, the specific gravity of the apophyllite has increased. Prehnite with the original gravity comparable to the substituted chlorite has not undergone a significant change in gravity.

X-ray powder patterns of the various pseudomorphs show dominant lines of the original minerals indicating that the degree of chlorite substitution is certainly less than 50%. A secondary optical examination disclosed that some of the crystals had undergone far less alteration than others, and although the unaltered portions were extremely minute, their optical properties were clearly those of the original minerals herein named. It would have been impossible to identify the substances directly by optical means, but with other data at hand the optical inspection proved fruitful.

Acknowledgments

The authors are particularly grateful to Mr. Stephen Ridel for his offering the specimens for study and for his patient waiting for results. They also wish to thank Mr. Robert A. Lancaster for his crystal drawings and Mr. Phokian Karas for his photographs of the specimens. We are also very grateful to the officers of the Materials Service, Inc. for the privilege of visiting the quarry.

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ERRORS IN POINT-COUNTER ANALYSIS

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The extension of modal analysis to sawn surfaces (Jackson & Ross, 1956; Plafker, 1956; Emerson, 1958; Fitch, 1959) increases interest in the effects of rock-coarseness, sample area and count length on precision. A comment on Dr. F. Chayes' recommendations with regard to these three factors is therefore offered.

In the course of his exhaustive study of point-counter technique, Chayes (1949; 1951; Chayes & Fairbairn, 1951) has clearly shown the presence of two sources of variance: one lies in using a point-count as a sample of a rock section, and the other lies in using a rock section as a sample of a specimen. In his most recent publication on the subject, Chayes (1956) sets out to compare the sizes of the two variance components but does not pursue this course to a conclusion (p. 89). It is the purpose of this note to suggest—

1. that Chayes' data do not oblige the abandonment of the original intention;
2. that the data do in fact make possible a comparison of the two variance components.

Chayes' analysis is founded on experimental estimates of the variance between a point-count and its parent rock specimen for twelve different samples. The variance estimates, V_p in Chayes' notation, thus contain the two variance components mentioned above, $V_{counting}$ and $V_{rock\ sampling}$. By using a fixed count length, $V_{counting}$ is maintained constant throughout the entire analysis; moreover its size is indicated by a great body of Chayes' earlier data. Thus the twelve estimates of V_p give by subtraction twelve estimates of $V_{rock\ sampling}$, in which the effects of sample area and rock coarseness may be studied.

The stumbling block in Chayes' analysis is the fact that two of the experimental estimates of V_p are *less* than the predicted value for $V_{counting}$ alone. Possible reactions to this discovery are—

1. the formula $V_p = V_{counting} + V_{sampling}$ may be rejected;
2. the value predicted for $V_{counting}$ from Chayes' earlier studies may be rejected, in favour of a smaller value.

The second possibility may be sub-divided into—

2a. a general modification of Chayes' estimate of $V_{counting}$;

2b. a postulate that on account of some quality of the particular rock from which the two anomalous results are derived, the predicted counting error is not generated in full.

It is considered that, independently of any measurements, the *design* of the test makes the first reaction unwarrantable.

Of the remaining possibilities, 2b is preferred. Each of the twelve estimates is based on thirty measurements, making 360 in all; but the evidence supporting Chayes' prediction for V_c is so great that it cannot be set aside by indirect indications even from 360 measurements. It is suggested alternatively that in the particular circumstances of the latest test, the conditions of randomness on which Chayes' prediction is based may not be fulfilled by one of the rocks. This last suggestion in only tentative pending experimental study; by contrast, it is considered that the rejection of the first possibility given above requires no empirical support, but is instead a necessary consequence of the experiment design.

A more constructive comment on Chayes' treatment may be made concerning comparable analyses on coarse and fine rocks. It is suggested that if IC determinations (Chayes, 1956, p. 72) show one rock to be twice as coarse as another, then if counts of equal length are made on each, but the point-spacing over the coarse rock is twice that over the fine one, then the analyses will have the same precision; in other words, that a wide grid over a coarse rock behaves simply as a scale-model of a close grid over a fine rock. If this is so, the area sampled and the rock coarseness need not be treated as independent factors, having separate effects on precision, but may be combined into a single factor.

In actual fact, clotting frequently distinguishes a coarse rock from a fine one. However study of a theoretical model and a test on Chayes' results both indicate that the divergence of coarse rocks from scale models of fine ones is not detectable within the spread of the counting and sampling errors. Consequently, it is recommended that V_s is treated not as a function of sample area and IC where area and IC are independent, but as a function of a single variant, area x IC^2; for this product is constant through any series of analyses which are scale models of each other, being related to the actual number of grains in the sample.

To summarize, it is suggested that instead of comparing data with independent equations $V_p=f(\mathrm{A})$ and $V_p=f(\mathrm{IC})$, and considering the influence of count length separately, all the factors may be included in a single relation—

$$V_p = V_c + V_s = f(\text{count length}) + f(\mathrm{A.\,IC}^2)$$

Further, accepting Chayes' finding that except in the anomalous case mentioned, V_c may be equated to $p(1\text{-}p)/n$, (p = proportion of mineral in rock; n = count length), it can be suggested from theory and verified from Chayes' data that the function $f(\mathrm{A.IC}^2)$ is of the form $k.(\mathrm{A.IC}^2)^{-1}$; for A in mm.^{2}, these data give $k=4.58\times10^6$. (Bayly, 1960)

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AN ASSOCIATION OF AWARUITE WITH HEAZLEWOODITE

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Awaruite, the naturally occurring alloy of iron and nickel, has been observed by Ramdohr (1950) in association with pentlandite and heazlewoodite in specimens from Trial Harbour, Tasmania. The awaruite in the specimens studied by Ramdohr was rare and extremely fine grained, and was identified by a process of elimination. Its identity could not be confirmed by etch, microchemical or *x*-ray examination.

Recently, during the development of the Lord Brassey mine at Heazlewood, north-western Tasmania, a seam of high grade heazlewoodite ore was encountered in a cross-cut adjacent to the main adit. Examination of heazlewoodite collected from this seam in May, 1959, has revealed an occurrence of awaruite intergrown with the heazlewoodite (Fig. 1).

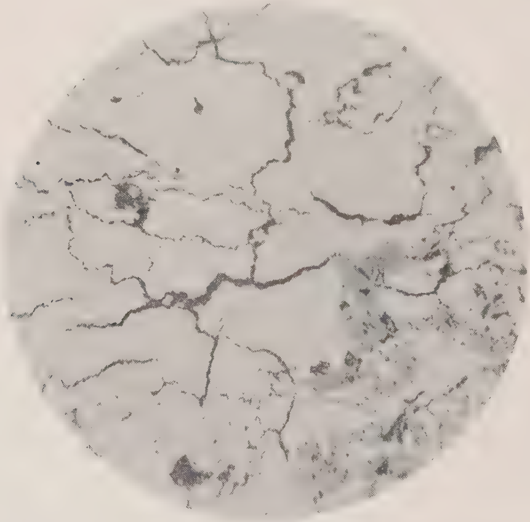

Fig. 1. "Net-like" intergrowth of awaruite (black, etched with 1:1 HNO_3 for 4 seconds) and heazlewoodite (white), with some magnetite (gray). Polished section ×150.

The awaruite is white in reflected light and slightly harder than the associated heazlewoodite. It is isotropic, with a reflectivity higher than that of the heazlewoodite (51.6–59.3 per cent; Williams, 1958) but lower than that of native copper (61–89 per cent; Uytenbogaardt, 1951), which occurs in minute amounts in some of the specimens. It polishes readily to a smooth, scratch-free surface.

The etch reactions of the awaruite were found to be as follows: HNO_3—etches rapidly black, with effervescence; HCl—negative; KCN—negative; $FeCl_3$—etches black almost instantaneously; KOH—negative; $HgCl_2$—variable, sometimes negative and sometimes etching slowly black. Positive microchemical tests for iron and nickel were obtained, though these were both open to some doubt because of the strong possibility of contamination with finely intergrown magnetite and heazlewoodite.

Strong adherence of the mineral powder to the needle of the solenoid-type microdrill used to obtain material for microchemical testing suggested that the awaruite is highly magnetic.

Awaruite from Awarua Bay, New Zealand, was found to give similar etch reactions. The statement by Uytenbogaardt (1951), after Schneiderhöhn and Ramdohr (1931), that awaruite is insensitive to all standard etch reagents is, therefore, incorrect. The possibility that the observed behaviour is anomalous, because of galvanic or other effects induced by the adjacent heazlewoodite, was considered but was rejected in view of the similar etching behaviour of the pure New Zealand awaruite. Ramdohr (1951) describes the etch behaviour of awaruite as "nicht untersucht" (not investigated).

X-ray powder diffraction photographs of the Heazlewood and New Zealand awaruites gave the lines listed in Table 1. Both resemble that of

TABLE 1. AWARUITE—(Ni, Fe); *X*-RAY POWDER DATA

Cubic, *Fm3m*.

Radiation: $CuK_{\alpha 1}$; $\lambda = 1.5405$ Å. Camera Diameter: 114.6 mm.

Heazlewood, Tasmania		hkl	Awarua Bay, New Zealand	
$d_{meas.}$ Å	$I_{est.}$		$d_{meas.}$ Å	$I_{est.}$
2.044	10	111	2.044	10
1.775	6	200	1.772	6
1.255	4	220	1.253	3
1.070	5	311	1.069	4
1.024	2	222	1.023	1
0.815	2b*	331	0.814	1b
0.793	2b	420	0.792	1b
a_0: 3.549±0.003 Å			3.545±0.003 Å	

* b denotes a broad line.

metallic nickel, although each has a slightly different unit cell parameter. The cell edge of the Heazlewood mineral was calculated from the observed spacings as 3.549±0.003 Å. Donnay and Nowacki (1954) record 3.525±0.001 Å for the cell edge of pure nickel; this corresponds to the value of 3.5171 "Å" (probably kX units) given by Owen, Yates and Sully (1937). The difference in cell parameters can probably be attributed largely to the presence in the awaruites of alloyed iron.

The Heazlewood awaruite occurs as fine, ramifying intergrowths with heazlewoodite and magnetite (Fig. 1); the net-like appearance of the intergrowth is frequently reminiscent of grain boundary segregations or replacements. Individual areas of awaruite seldom exceed 0.3 mm. in diameter.

Because the awaruite is so fine grained and is present in such small proportions, its chemical composition could not be determined by direct chemical analysis. Its magnetic character could not be used to prepare a concentrate because of the presence of relatively large proportions of magnetite; in any event the intergrowths are so fine that mechanical separation of the awaruite from the heazlewoodite and magnetite would be extremely difficult.

However, comparison of its measured unit cell parameter with the data of Owen, Yates and Sully (1937), who determined the variation in cell parameter for the series Fe—(Fe,Ni)—Ni, suggests a composition within the range 77–82 per cent nickel.

As a check on the reliability of such an estimation, the New Zealand awaruite was examined further, since it was available in quantities sufficient for chemical analysis. The cell edge of this awaruite, 3.545±0.003 Å suggests a composition within the range 80–85 per cent nickel; however a chemical analysis (Table 2), made on a hand-picked sample of 0.25 g.,

Table 2. Chemical Analyses

	1	2	3
Fe	27.02	27.34	25.2
Ni	69.06	69.88	70.2
Co	2.75	2.78	4.0
Cu	—	—	0.3
Insol.	1.28	—	—
	100.11	100.00	99.7

1. Awaruite, Heazlewood, Tasmania—analyst: P. J. Sinnott.
2. (1), after removal of insolubles and recalculation to 100 per cent.
3. Nickel-iron, Eastern Townships, Quebec (Nickel, 1958).

showed only 69.8 per cent nickel. This lack of agreement can possibly be attributed, at least in part, to the unknown effect on the parameter of the presence of extraneous elements, notably cobalt. The New Zealand awaruite contains 2.78 per cent cobalt.

A similar, but smaller, discrepancy is apparent in the results of Nickel (1958), who found $a_o = 3.551–3.555$ Å for awaruite (nickel-iron) from Quebec, suggesting, from the data of Owen, Yates and Sully, a nickel content of 75–78 per cent. Chemical analysis, however, showed only 70.2 per cent nickel, together with 4.0 per cent cobalt.

It is thus apparent that awaruite compositions estimated from cell parameters in this way are approximations only, with wide limits of error.

Some published figures for the unit cell parameters of awaruite (Ramdohr, 1951) and nickel-iron (Palache, Berman and Frondel, 1944) agree on 3.59 Å, a figure significantly different from those obtained by the author and by Nickel (1958). However, the former determinations appear to have been made on meteoritic taenites, with much lower nickel contents of 30–40 per cent. In this event they agree fairly well with the results of Owen, Yates and Sully, who obtained $a_o = 3.577$ kX for an alloy containing 30 weight per cent nickel, and 3.590 kX for one with 40 weight per cent nickel (both figures originally quoted as Ångstrom units). A general figure of 3.54–3.55 kX (i.e. 3.55–3.56 Å) seems more appropriate for terrestrial awaruite.

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LAWSONITE METAGRAYWACKES IN NEW ZEALAND

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Lawsonite is a wide-spread mineral in low grade geosynclinal metamorphic rocks and is especially well known from various points in the circum-Pacific orogenic belt. Hitherto however it has not been recorded from rocks of the New Zealand Geosyncline.

The writer has recently found that lawsonite is constantly present in a belt, over two miles wide, of slates and volcanic graywackes of the Maitai Group (Permian) exposed in the Maitai Valley, Nelson, New Zealand. In spite of distinct slaty cleavage, delicate sedimentary structures are clearly preserved and from hand specimens perhaps few geologists would regard these rocks as metamorphic. Yet internal reconstitution is far advanced, and the rocks may be classified as lawsonite metagraywackes and slates. They contain the mineral assemblage albite-chlorite-lawsonite-quartz-calcite-muscovite with varying proportions of the constituents. Lawsonite marble has also been found. The lawsonite occurs as tablets, 0.1–0.01 mm. long, with typical transverse fractures, $\alpha 1.662$, $\beta 1.672$, $\gamma 1.684$. The tablets occur both in albitized plagioclase and in the matrix. These lawsonite metagraywackes recall the jadeite metagraywackes of the Franciscan group reported by Bloxam (1956) and McKee (1958), and like these are of regional extent. McKee found that the albite-lawsonite assemblages develop before jadeite-lawsonite-quartz or glaucophane during progressive alteration, a conclusion which accords with the occurrence of albite-lawsonite assemblages in Indonesia (de Roever, 1947) and elsewhere.

Jadeite and glaucophane have not yet been found in the Nelson district. Instead the known part of the lawsonite-bearing belt, which is at least 17 miles long, appears to pass at its southern end into prehnite—and pumpellyite-bearing assemblages of the prehnite-pumpellyite metagraywacke facies. Typical representatives of this new facies have been described elsewhere (Coombs, *et al.*, 1959) and it has been defined (Coombs, 1959) to include those assemblages produced under physical conditions in which the following are commonly formed: quartz-prehnite-chlorite, or quartz-albite-pumpellyite-chlorite, without zeolites and without the characteristic minerals of the glaucophane schist facies, jadeite or lawsonite. Similarly on its east side, the lawsonite belt is separated by some comparatively little-altered rocks containing spasmodic prehnite, from prehnite-bearing volcanic metagraywackes, tuffs and metavolcanics of the Te Anau group. It is in the Te Anau Group that the ultramafic rocks of the Dun Mountain belt have been emplaced.

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THE AMERICAN MINERALOGIST, VOL. 45, MARCH–APRIL, 1960

FLUOCERITE AND ASSOCIATED MINERALS FROM THE BLACK CLOUD PEGMATITE, TELLER COUNTY, COLORADO*

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The Black Cloud pegmatite, which is exposed 800 feet north of U. S. Highway 24, 2.3 miles west of Divide in Teller County, Colorado, was studied in June 1957 and June 1958. Laboratory studies were completed between September 1958 and May 1959. Both field and laboratory work were supported by a grant from the Michigan Memorial-Phoenix Project Fund, Project 150. This study is part of a continuing investigation of the pegmatites of the South Platte–Pikes Peak area of Colorado, to which field work during the last three summers has been devoted (Heinrich, 1958), and represents an extension of a previous reconnaissance study of rare-earth pegmatites in Colorado (Heinrich, 1948).

The lensoid pegmatite, well exposed in two large open cuts, trends N. 40° W. with generally steeply dipping to vertical contacts with Pikes Peak granite. It is nearly 350 feet long, and at its bulbous northwestern nose it is 110 feet thick. The sharply defined internal structure includes:

1. Wall zone (quartz, oligoclase, microcline) as much as 25 feet thick, fine grained, with much graphic quartz-oligoclase rock.
2. Intermediate zone, 50–75 feet thick, masses of pink microcline with interspersed pods of white to gray quartz and scattered small beryl crystals.
3. Core of milky quartz, as much as 50 feet thick, studded with primary vugs 1–6 inches across enclosing clear, milky or smoky quartz crystals.

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* Contribution No. 235 from the Department of Mineralogy, The University of Michigan, Ann Arbor.

4. Irregular replacement lenses and pods, 2–6 feet long, localized chiefly along contacts between quartz and microcline masses in the intermediate zone.
5. Fracture-controlled concentrations of biotite in books as large as 2 feet across.

The replacement bodies (0.1–0.5% of the pegmatite volume) are of two types: fluoritic (more common) and albitic. They contain nearly all the unusual minerals.

The accessory mineral assemblage is characterized by fluorides, phosphates, multiple oxides, and silicates of both Ce- and Y-group minerals (Table 1). Those occurring almost exclusively in the fluoritic pods are xenotime, monazite, fluocerite, gadolinite, and thorite. Samarskite is confined to albitic pods. The other accessory species occur in both types, as does purple fluorite (Type III). Glassy green (Type I) and porcelanoid gray-tan (Type II) fluorites occur in fluorite-rich pods only. The bulk of the monazite and xenotime occurs as microscopic inclusions in Type II fluorite (Col. A, Table 3). The occurrence of yttrotantalite is the second authenticated for the United States; it was found as small grains in microcline.

Table 1. Accessory Minerals of the Black Cloud Pegmatite

Fluorides	Fluorite, fluocerite
Phosphates	Monazite, xenotime
Multiple oxides	Samarskite, yttrotantalite, fergusonite
Silicates	Allanite, gadolinite, thorite, beryl, bertrandite

Fluocerite, which has been reported previously in the United States only from Cheyenne Mountain, El Paso County, Colorado (Allen and Comstock, 1880; Hillebrand, 1899), occurs in very modest amounts in the pegmatite. It was found only on the dump in pieces 2 to 8 inches long. These specimens, which are aggregates of fluocerite, monazite, usually with some feldspar and several other minerals, have an extraordinary high specific gravity and are markedly radioactive. They are dark olive brown in color with a dull waxy luster. They show a characteristic texture consisting of ovoids of medium brown fluocerite as much as 0.8×1.0 inches in section, surrounded by a fine-grained rim which varies in color from darker brown to black to red (Fig. 1). The red marginal mineral is monazite. The other interstitial species has not been certainly identified (Unknown Mineral No. 1).

Optical properties of the fluocerite are as follows: uniaxial negative, indices of refraction $\omega = 1.615$, $\epsilon = 1.607$. The specific gravity is 5.80, by means of the Berman microbalance. This value is low owing to inclusions, chiefly monazite.

In thin section the mineral shows narrow multilamellar twinning,

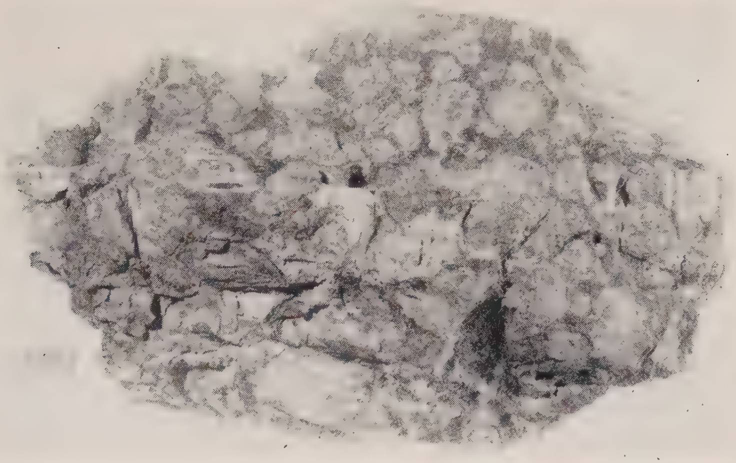

FIG. 1. Largest mass of fluocerite (9×14 cm.) found at Black Cloud pegmatite, Teller County, Colo. Irregular to rounded areas are fluocerite; dark interstitial material is chiefly unidentified RE silicate; darker brown areas in lower left are monazite.

pyramidal in orientation, making an angle of 45° with the trace of the perfect (0001) cleavage. Numerous inclusions of various types are present: 1) small specks or irregular blebs of monazite; 2) rounded grains of a high relief, turbid, golden brown isotropic species which, from the radial cracks that surround them, is strongly radioactive; and 3) aggregates of pinpoint flakes of a brilliant crimson mineral which shows very slight pleochroism, has low birefringence and moderate relief. The centers of the fluocerite ovoids are usually markedly altered to a yellowish brown, very fine-grained constituent, whose formation appears to have been controlled somewhat by the basal cleavage. It is apparently isotropic, and its segregation may have resulted from the selective replacement of cores of originally zoned fluocerite crystals.

The abundant associated monazite occurs as small blebs and marginal rims that extend thin veinlets into the fluocerite, especially along the cleavage.

Most of the other interstitial material is an exceedingly fine-grained aggregate, brownish to grayish brown in color, which shows very low birefringence and appears to have undergone extensive structural damage as the result of metamictization (Unknown Mineral No. 1). Some of this substance appears to be granular, some of it pseudomorphous after a bladed species. Most of it is barely translucent in thin section and has a mean index of refraction, $n=1.81$. Spectroscopic analysis indicates it is probably a silicate principally of Al, Fe, Ca, La and Nd. We have not

TABLE 2. *X*-RAY DATA FOR FLUOCERITE

A		B	
d	I/I_1	*d*	*I*
3.629	.7	3.64	m
3.536	.6	3.55	m
3.186	1.0	3.19	vs
2.534	.3	2.54	vw
2.046	.8	2.05	s
1.997	.8	2.00	vs
1.813	.2	1.815	vw
1.780	.7	1.782	m
1.720	.5	1.723	w
1.614	.2		
1.592	.1		
1.429	.4	1.427	w
1.357	.4	1.36	w
1.342	.3	1.343	w
1.317	.4	1.32	w
1.268	.1		
1.256	.1		
1.209	.1		
1.180	.2		
1.170	.4	1.175	w
1.144	.1		
1.121	.4	1.127	vw
1.076	.1		
1.041	.4	1.045	vw
(and others)			

Col. A. (Ce, La)F_3 (synthetic). ASTM II—1152.
Col. B. Fluocerite, Black Cloud pegmatite, Colo.

TABLE 3. ANALYSES BY *X*-RAY FLUORESCENCE

	A	B		A	B
Ca	30.3	0.5	Gd	0.21	0.1
Y	1.8	0.2	Dy	0.13	
La	0.3	10.8	Er	0.08	
Ce	0.8	19.0	Fe	0.3	
Pr	0.25	1.1	Th	0.022	0.3
Nd	0.7	3.6	Sr	0.014	
Sm	0.1	0.3	Mo	0.5	

A. Type II fluorite, Black Cloud pegmatite, Colo., containing abundant microscopic inclusions of xenotime and monazite.
B. Fluocerite, Black Cloud pegmatite, Colo., containing about 8% quartz as inclusions.

been able to match its x-ray pattern with that of any other species available to us.

Other minerals commonly associated with the fluocerite are small grains of altered gadolinite, small grains and crystals of metamict thorite, and a rare unidentified colorless species of moderate birefringence, biaxial character, with a mean index of refraction near 1.54. Relict in sections rich in fluocerite are corroded grains of quartz, plagioclase, and microcline-perthite.

The identity of the fluocerite was checked by means of an x-ray powder photograph (Table 2); its composition is given in Table 3, Column B.

Most of the rare-earth pegmatites of the South Platte–Lake George area are enriched in Y-group rare earths. The nearest neighbor to the Black Cloud, the Teller pegmatite near Lake George (Glass *et al.*, 1958), as well as most of the other pegmatities of the district, are characterized by Y-earth accessory mineral assemblages. The Black Cloud deposit appears to be unique for the area in having an accessory mineral group of both Y- and Ce-group elements.

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DETRITAL EUXENITE AND ASSOCIATED MINERALS, SAND BASIN, GRANITE COUNTY, MONTANA*

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The presence of metamict multiple oxide and silicate minerals ("radioactive blacks") in many stream placers in west-central Idaho has long been established (e.g. Shannon, 1922), and in 1952–1954 the geology of these deposits was studied in detail (Mackin and Schmidt, 1956; summarized in Heinrich, 1958). The radioactive placer minerals have been derived chiefly from the quartz monzonitic facies of the Idaho batholith, in which they occur locally as accessory species. "Segregations" of quartz

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* Contribution No. 236 from the Department of Mineralogy, The University of Michigan, Ann Arbor.

TABLE 1. MINERALS OF THE HEAVY FRACTION OF THE BLACK SANDS OF SAND BASIN, MONTANA

A. Non-radioactive species (in approximate order of abundance)

Major	1. Magnetite, usually in lustrous euhedral octahedra with common re-entrant angles parallel to (101). Up to 90% of some samples
	2. Ilmenite, somewhat rounded
	3. Garnet, red, translucent, euhedral, trapezohedra
	5. Epidote
	5. Leucoxene, well rounded grains
Minor	6. Hornblende
	7. Biotite, in part chloritized
Trace	8. Kyanite
	9. Rutile
	10. Tourmaline
	11. Limonite, pseudomorphous after pyrite cubes

B. Radioactive species (in order of abundance)

1. Sphene. Yellow-brown translucent euhedra of the "envelope" type, flattened parallel to (001) and bounded by {100}, {112}, and {001}; some crystals of prismatic habit also present, 0.5–2 mm. in size (Fig. 1, B). Minute black opaque inclusions common; fractures abundant. Fluoresces olive green under unfiltered ultraviolet from high pressure mercury arc. Very weakly radioactive.
2. Monazite. Reddish-yellow tabular euhedra flattened parallel with (100), with a general rhombic outline. Usually smaller than sphene, 0.25–1 mm. in size (Fig. 1, C). Fluoresces emerald green. Radioactivity moderate, stronger than euxenite.
3. Euxenite. Submetallic. Broken prismatic crystals. Common forms {110}, {010}, and {111}. 0.5–2 mm. in size. Two types of alteration, 1) (more common) a tan powdery fracture filling, and 2) milky to bluish white coating on unabraided surfaces (Fig. 1, A). Radioactivity weak.
4. Allanite. Dull black irregular grains and prismatic crystals, 0.5 mm. in size. Subangular to subrounded with tan alteration films. Strongly radioactive.
5. Zircon. Very slender elongate prismatic euhedra, colorless and free of inclusions; 0.25 mm. or less in length. Feebly radioactive.
6. Thorianite. Translucent deep emerald green grains of irregular outline. 0.25–0.5 mm. in size. Very strongly radioactive. *X*-ray powder pattern of unignited grains showed two pronounced diffuse bands from 7.5–2.6 and from 2.2–1.8 Å.

monzonite containing marked concentrations of these minerals range in size from a few inches to a few tens of feet and are megascopically identical with enveloping quartz monzonite that has a much lower content of these species.

In some areas deep pre-Wisconsin regoliths provided initial concentrations, and subsequent placer accumulations have resulted from late Pleistocene glacial derangements of the drainage or drainage blocking by Pleistocene lava flows (Mackin and Schmidt, 1956). The percentages of metamict species decrease very rapidly downstream.

In 1954 Mr. Joseph Abbey discovered "radioactive blacks" in stream sands in Sand Basin, a small park-like area in Granite County, Montana, embracing parts of the drainages of Sand Basin, Bowles and West Fork Rock Creeks, all tributaries of North Fork Rock Creek. The deposits lie at an elevation of about 6400 feet, on the east side of the Sapphire Mountains, about 5 miles southeast of Skalkaho Pass. The U. S. Bureau of Mines sampled the deposits in 1954 by means of about 40 shallow pits. The senior writer studied the deposits in July 1957, and laboratory studies, supported by a grant from Michigan Memorial-Phoenix Project No. 150, were completed in June 1959. The deposits are of interest not only because of their unusual mineralogy but also because they are the first major find of "radioactive blacks" in Montana placers, thus representing the easternmost known occurrences of such deposits associated with rocks of the Idaho batholith.

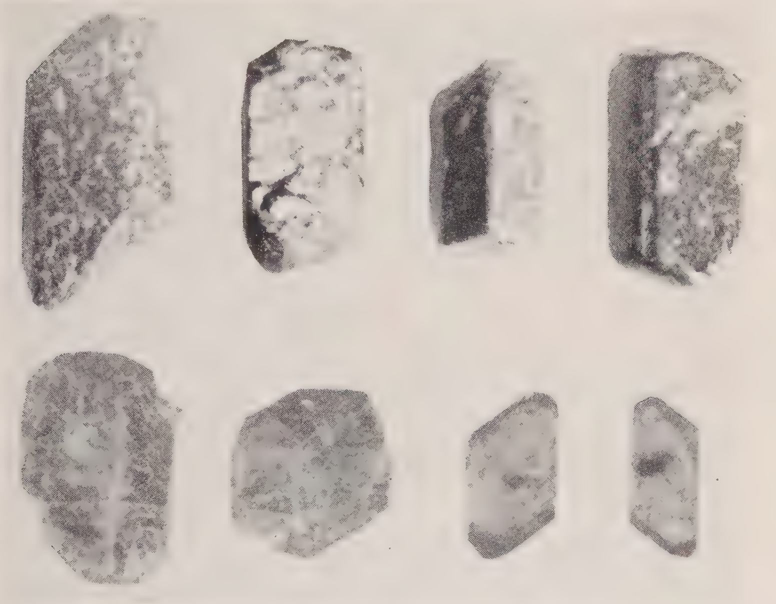

FIG. 1. Chief radioactive detrital minerals of Sand Basin placer deposits, Granite County, Montana. A. (top row) Euxenite; note zoning in second from left (×15). B. (lower left) Sphene (×15). C. (lower right) Monazite (×30).

The area is underlain by a coarse-grained granodiorite assigned to the Idaho batholith, which just to the north intrudes rocks of the Newland formation of the Precambrian Belt Series. The granodiorite crops out as low rounded ridges that separate the sand-filled basins ("flats"). Where the ridges slope down to constrictions or terminations of the flats, a jumble of granite boulders is a characteristic feature. The area does not appear to have been glaciated (Alden, 1953) but is periglacial. The streams of the "flats" are irregular in direction, meandering, and incised as much as 7 feet. In contrast, the upper part of West Fork Rock Creek, whose course is not marked by flats, is straight and its tributaries appear to have been controlled partly by jointing in the granodiorite.

The valley fill of the "flats" consists of a locally developed veneer of subangular rock pieces, 8 inches or less in size, mainly of quartzites, nodular muscovite schist, and other low-grade metamorphic rocks that are foreign to the immediate area of the deposits. Many of the finer-grained accompanying particles of this surficial layer are of weathered granodiorite or its constituents. In stream banks and pits the bulk of the valley fill can be seen to consist of coarse sand that locally contains layers and lenses of black minerals, usually one inch or less thick. Diamond drilling has shown that this sand continues to a depth of at least 35 feet in the central part of Sand Basin, but grades downward into a silt locally as much as 55 feet thick, which is much poorer in heavy (and radioactive) detritals.

TABLE 2. MINERALS OF IDAHO-MONTANA "RADIOACTIVE BLACKS"

Monazite, sphene, euxenite, allanite, zircon, thorianite	Sand Basin, Mont.	This paper
Monazite, xenotime, thorite, samarskite, betafite, brannerite, davidite, euxenite	Red River Valley, Elk City, Idaho	Armstrong and Weis (1957)
Monazite, sphene, zircon, samarskite, columbite, polycrase	Idaho City and Centerville, Idaho	Shannon (1922)
Monazite, xenotime, columbite, samarskite, fergusonite, zircon	Dismal Swamp, Elmore Co., Idaho	Shelton and Stickney (1955)
Monazite, xenotime, columbite, euxenite, allanite, sphene, zircon	Bear Valley, Valley Co., Idaho	Mackin and Schmidt (1956)
Thorite, sphene, allanite, zircon	Hailey, Blaine Co., Idaho	Mackin and Schmidt (1956)

The minerals of the black sands are listed in Table 1. The concentrates, panned in the field, were studied as follows: 1) Separation of the heavy minerals in methylene iodide. 2) Removal of the magnetite. 3) Fixing the remaining optically unidentifiable grains on glass plates with diluted Duco cement, and laying over these mounts x-ray film to produce autoradiographs. 4) Removal of individual radioactive grains for powder x-ray study (following ignition, if necessary). The euxenite, allanite and thorianite identities were checked via x-rays.

A specimen of granodiorite from the north side of Sand Basin was crushed and the concentrated heavy mineral fraction was found to contain magnetite, ilmenite, sphene, monazite, euxenite, allanite, thorianite, and zircon. The size, habit, color, fluorescence, and radioactivity of these minerals are essentially identical to these properties as exhibited by the same species in detrital form. Thus of the abundant heavy species in the sands, only garnet and epidote have come from outside of area underlain by granodiorite.

Table 2 compares "radioactive black" suites from some of the Idaho and Montana placers, and in Table 3 are listed similar suites that have been identified *with certainty*, as occurring as accessory minerals in nonpegmatitic rocks of the Idaho batholith. Armstrong and Weis (1957, p. 35) state, with respect to deposits of the Red River Valley, Idaho, that "The source of the uranium-bearing minerals is not known, but because they characteristically occur widely disseminated in pegmatites, it is assumed that pegmatites related to the Idaho batholith are the source of the radioactive blacks in the placer gravels." Certainly some pegmatites of Idaho batholith affinity which are known to contain some of these "radioactive blacks" may well be an additional source for them in some areas, but the evidence is now preponderant that many of these deposits are of nonpegmatitic derivation.

TABLE 3. ACCESSORY MINERALS OF ROCKS OF IDAHO BATHOLITH

Sphene, monazite, euxenite, allanite, thorianite, zircon	Sand Basin, Mont.	This paper
Monazite, zircon, sphene, allanite, xenotime, thorite	Idaho batholith in general	Larsen and Schmidt (1958)
Euxenite, monazite	Bear Valley, Idaho	Mackin and Schmidt (1956)
Thorite, sphene	Hailey, Idaho	Mackin and Schmidt (1956); Robertson and Storch (1955A, B)

The Idaho batholith has several outliers that extend eastward into Montana, just as the Boulder batholith has outliers to the west of its main mass. The assignment of outliers in this "intermediate zone" to one or the other of these intrusive units has been fraught with considerable uncertainty. Since it is now known that units of the Idaho batholith contain a widespread varied assemblage of U-Th-Nb-Ta species as accessory constituents and since no similar assemblages, either in igneous rocks or derived placers, are reported for the Boulder batholith, this relationship might offer an additional characteristic for correlating these intrusive units.

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HYDROXYL ION CATALYSIS OF THE HYDROTHERMAL CRYSTALLIZATION OF AMORPHOUS SILICA; A POSSIBLE HIGH TEMPERATURE pH INDICATOR

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INTRODUCTION

A study has been made of the effect of hydroxyl ion concentration on the time for the formation of quartz from silicic acid at 245° C. The time appears to be almost a linear function of reciprocal hydroxyl ion concentration. It is considered that all observations on this crystallization can be explained by a mechanism involving the concentration of a singly charged silicate anion as the rate controlling species. The effect of other mineral substances, in particular silicates, on this reaction may provide a method of determining the extent of hydrolysis of these minerals at moderate temperatures.

Previously a number of workers have found that when amorphous silica is crystallized under hydrothermal conditions, a number of intermediate phases tend to develop before the stable phase quartz is formed. Corwin et al. (1953) have shown that in a fixed time either cristobalite or quartz is produced, quartz being favored by high pH. Carr and Fyfe

(1958) studied the effect of pressure, and to a less extent temperature, on the process in pure water and found that the reaction was much more sensitive to pressure changes than temperature and that quartz formed only after the appearance of both cristobalite and silica-K (keatite). They considered that the rate was controlled basically by induced nucleation and that the pressure sensitivity might by explained with reference to the effect of pressure on the dissociation of silicic acid.

The present experiments were designed to obtain more data on the effect of hydroxyl ion concentration so that some definite selection of the rate controlling species might be possible.

EXPERIMENTAL

Small stainless steel test-tube bombs with a capacity of 1.5 ml. were used. Ignited silicic acid was sealed in a silver tube with 0.1 ml. of carbonate free sodium hydroxide. The amount of silica used was always sufficient to maintain a molar ratio of silica to hydroxide of the order of one hundred. This silver tube was placed in the bomb and sodium hydroxide solution added on the outside to balance the pressure in the tube. The pressure in the vessel must be nearly that of pure water at 245° C.,

TABLE 1. PRODUCTS OF CRYSTALLIZATION OF AMORPHOUS SILICA

NaOH Concentration	Time (Hours)	Products	
		Cristobalite	Quartz
0.100 Molar	15	poor	—
0.100 Molar	16	poor	v. weak
0.100 Molar	17	poor	weak
0.100 Molar	18	poor	weak
0.100 Molar	19	—	almost 100%
0.100 Molar	20	—	100%
0.017 Molar	20	poor	—
0.017 Molar	40	poor	—
0.017 Molar	70	weak	—
0.017 Molar	80	strong	weak
0.017 Molar	90	weak	strong
0.017 Molar	100	—	almost 100%
0.017 Molar	110	—	100%
0.012 Molar	50	poor	—
0.012 Molar	70	weak	—
0.012 Molar	90	strong	—
0.012 Molar	120	strong	weak
0.012 Molar	130	V. strong	weak
0.012 Molar	140	—	100%

i.e. 36 bars. The starting material, powdered silicic acid (Mallinckrodt Chem. Co.) was ignited at 700° C. for 24 hours before use. This was found necessary, as otherwise water from silicic acid dilutes the alkali during the course of the run. *X*-ray patterns indicated that no detectable change in structure of the material occurred with this heating and the broad hump in the region of the strong cristobalite line remained little altered. Identification of all phases formed was by means of a Phillips *x*-ray diffractometer.

RESULTS AND DISCUSSION

Experiments were carried out at three hydroxyl ion concentrations to find the minimum time for complete formation of quartz. Obviously, a greater range of concentrations was difficult to use as times were either too short or inconveniently long. A summary of critical results from some 56 runs is given in Table 1. With short time runs, normally at least four were carried out and the behavior was reproducible to within one hour.

The results in the above table support a number of conclusions that may have bearing on the mechanism of the reaction.

1. Quartz does not appear until cristobalite has first been formed. How well the cristobalite develops depends on the strength of the sodium hydroxide used, and hence on the length of the run.
2. When cristobalite is well developed, quartz is usually present.
3. Tridymite and silica-K were not formed in sufficient quantity to show on an *x*-ray pattern.
4. The change cristobalite to quartz is much more rapid than the reaction amorphous silica to cristobalite.

Analysis of the above results indicates, that within the experimental uncertainty, the time for 100% formation of quartz is a linear function of 1/[NaOH]. For each strength of sodium hydroxide it is possible to estimate the concentrations of various possible reacting species present, in particular $Si(OH)_4$, OH^-, $H_3SiO_4^-$ and $H_2SiO_4^{--}$. In this range experimental values of K_w are available from Noyes and Kato (1907). Values for the first and second dissociation constants of silicic acid were taken from Greenberg and Price (1957) and Harman (1927). These latter values were extrapolated to 245° C. using a relation suggested by Pitzer (1937) which appears to hold fairly well to temperatures near 250° C. but is not adequate at higher temperatures (Ellis and Fyfe, 1957). Data on the solubility of quartz and amorphous silica were taken from Kennedy (1950). At 250° C. a saturated solution of amorphous silica has a concentration of 2.1×10^{-2} molar while a solution saturated with respect to quartz is 8.3×10^{-3} molar. The solutions used are always saturated with silica and Alexander, Heston and Ihler (1954) have shown that in such

TABLE 2. ESTIMATED CONCENTRATIONS OF IONIC SPECIES IN SILICA-SODIUM HYDROXIDE SOLUTIONS

[NaOH] (Initial)	0.100	0.017	0.012
$[H_3SiO_4^-]$	0.100	0.017	0.012
$[OH^-]$	1.5×10^{-2}	2.5×10^{-3}	1.8×10^{-3}
$[H_2SiO_4^{--}]$	2.3×10^{-4}	6.0×10^{-6}	3.1×10^{-6}

solutions the dominant species are $Si(OH)_4$ and $H_3SiO_4^-$. The hydroxyl ion concentration is given by the expression

$$[OH^-] = K_w[H_3SiO_4^-]/K_{a1}[Si(OH)_4]$$

and the concentration of $H_2SiO_4^{--}$ by the expression

$$[H_2SiO_4^{--}] = K_{a1}K_{a2}[Si(OH)_4][OH]^2/K_w^2.$$

It is assumed that the concentration of $H_3SiO_4^-$ is the same as the added sodium hydroxide. As the silicic acid is converted to quartz, the concentration of $Si(OH)_4$ in solution will decrease and the concentrations listed in Table 2 refer to initial experimental conditions. Using the concentrations in Table 2 and the experimental time for the 0.017 molar sodium hydroxide solution it is possible to predict the times required at the other concentrations. These predicted values are compared with the experimental values in Table 3. It is apparent that either OH^- or $H_3SiO_4^-$ could be rate controlling.

CONCLUSIONS

The results of crystallization in alkaline solutions indicate that either OH^- or $H_3SiO_4^-$ is the rate controlling species in the reaction. It might also be argued that the total amount of silica in solution determines the rate. This latter possibility can probably be eliminated by consideration of the pressure sensitivity in pure water found by Carr and Fyfe (1958) which is of a different order of magnitude to the effect of pressure on the solubility. Further, that hydroxyl is rate controlling can also be eliminated from consideration of the pressure effect. As silicic acid is a stronger acid than water and as the dissociation will increase with pressure (Ellis and Fyfe, 1957) then the concentration of hydroxyl in a pure water solu-

TABLE 3. ESTIMATED QUARTZ CRYSTALLIZATION TIMES

Strength of NaOH solution	0.100	0.012
Time if $[OH^-]$ rate controlling	17	140
Time if $[H_2SiO_4^{--}]$ rate controlling	3	190
Time if $[H_3SiO_4^-]$ rate controlling	17	140
Experimental values	18	140

tion should decrease with increasing pressure. It should be emphasized that both these arguments are only qualitative for they do not take into account changes in the rate constant with pressure and as yet data do not allow a sufficiently accurate analysis to completely settle the argument. It does appear, however, that all results can be reasonably explained if it is assumed that the rate is proportional to the concentration of $H_3SiO_4^-$ or a singly charged silicate anion. Goto (1955) has found that hydroxyl ions accelerate the polymerization of silicate ions in solution and it is not surprising that the present process involves similar factors.

From silica crystallization studies two features which have some general bearing on hydrothermal studies emerge. First, if the time for formation of quartz from a given type of silicic acid or amorphous silica is a measurable function of hydroxyl ion activity, then this time can be used as a pH indicator in more complicated environments. Chemists have long used catalytic effects of hydrogen ions as pH indicators in systems where chemical complications exclude the use of more direct techniques. As yet, we certainly do not have any simple method of measuring pH at elevated temperatures directly. It is planned to explore this aspect of the work in the very near future. It is hoped that it will be possible to obtain some information on the hydrolysis of complex aluminosilicate anions. Second, the results show clearly that it is possible to catalyze silicate reactions and to reduce the time for attainment of equilibrium by factors of the order of hundreds and thousands. At low temperatures it is a most difficult problem to obtain anything approaching equilibrium in silicate systems (e.g. Coombs et al., 1959) and it appears that serious consideration should be given to the mechanism of some more complex crystallizations so that possibilities of catalysis are fully appreciated.

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THE AMERICAN MINERALOGIST, VOL. 45, MARCH–APRIL, 1960

IMMERSION OILS WITH INDICES OF REFRACTION FROM 1.292 TO 1.411

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The authors and coworkers at the Oak Ridge National Laboratory have found that mixtures of perfluorotributylamine $(C_4F_9)_3N$, and polymers of chlorotrifluoroethylene $Cl\text{-}[CF_2\text{-}CFCl])_n\text{-}Cl$, have properties which make them quite suitable for use as immersion oils for index of refraction measurements in the range 1.292 to 1.411. These commercially available end members are colorless, odorless, and are miscible in all proportions. Their mixtures maintain the same indices of refraction (within one part per thousand) over many months, and possess viscosities and vapor pressures such that they are easy to handle. They have been found not to etch microscope slides, coverglasses, container bottles, or refractometer prisms to any visible extent.

Pertinent physical properties for perfluorotributylamine, a mixture of the polymers of chlorotrifluoroethylene known as KEL-F$^{(R)}$ Polymer Oil No. 10, and the fifth member of the polymer series of chlorotrifluoroethylene $[Cl\text{-}(CF_2\text{-}CFCl)_5\text{-}Cl]$ are tabulated in Table 1. The last ma-

TABLE 1

Material	Refractive Indices[a,b,c] n_D at 25° C.	Temperature Coefficients[a]		Vapor Pressures[b,c] mm. of Hg at 25° C.
		$-dn/dt$ (° C.)	Temperature range (° C.)	
$(C_4F_9)_3N$	1.292±0.001	0.00041	19–25	0.3
KEL-F$^{(R)}$ Polymer Oil No. 10	1.411±0.001	0.00033	22–51	3×10^{-4}
$Cl\text{-}(CF_2\text{-}CFCl)_5\text{-}Cl$	1.406±0.001	0.00034	25–42	2×10^{-3}

[a] Measured at the Oak Ridge National Laboratory by the authors with an Abbe Refractometer.

[b] Technical Bulletin on Inert Liquid FC-43. Minnesota Mining and Manufacturing Company.

[c] Technical Bulletin on Halofluorocarbons, Oils, Waxes, Greases, and Alkanes. Minnesota Mining and Manufacturing Company.

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* Operated for the U. S. Atomic Energy Commission by the Union Carbide Corporation.

terial has a lower viscosity than the mixture of polymers. Thus combinations of the labile perfluorotributylamine and the polymer mixture are somewhat more suitable for the lower part of the refractive index range while combinations of perfluorotributylamine and the pure Cl-(CF_2-$CFCl)_5$-Cl are better in the upper part of the refractive index range.

These oils have been used extensively at the Oak Ridge National Laboratory and at the Mound Laboratory for phase characterization and identification (1–4). The authors have observed no evidence of solubility or chemical reaction between these oils and the compounds described in the references.

No toxic or anesthetic effects for these materials have been observed (5–8) although they may cause skin irritation. Only at elevated temperatures (∼315° C.) do the polymers of chlorotrifluoroethylene decompose with production of toxic products (6–8).

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Dr. Arthur L. Day, retired director of The Geophysical Laboratory, died March 3, 1960, at the age of 90.

Professor Ciro Andreatta, of the University of Bologna, died February 6, 1960. He was a fellow of the Mineralogical Society of America.

Following a stroke suffered on November 23, Burnham S. Colburn died on December 26 at the age of 87, at his home in Biltmore, North Carolina. A banker by profession, Mr. Colburn was well known as an amateur mineralogist. He joined the society in 1930 and was elected to fellowship in December 1936. A memorial will appear in a later issue.

Dr. Richard H. Jahns, professor of geology and executive officer of the Department of Geology at the California Institute of Technology, has been appointed Chairman of the Division of Earth Sciences at The Pennsylvania State University. Dr. Jahns succeeds Dr. O. Frank Tuttle, who has been made Dean of the College of Mineral Industries at that institution.

9TH ANNUAL CONFERENCE ON APPLICATIONS OF *X*-RAY ANALYSIS

This conference will be held August 10–12, 1960, at the Stanley Hotel, Estes Park, Colorado.

For information, write Wm. Mueller, Head,
Metallurgy Division,
Denver Research Institute,
University of Denver,
Denver 10, Colorado.

FIFTH IUCR CONGRESS AND GENERAL ASSEMBLY

The fifth general assembly and international congress of the International Union of Crystallography will be held in Cambridge, England, August 15–20, 1960. Following the congress two symposia will be held (August 22–24) as follows: I. Thermal Motion in Crystals and Molecules. II. Lattice Defects and the Mechanical Properties of Solids.

For information write Dr. R. E. Rundle, Chemistry Department, Iowa State University, Ames, Iowa, or Dr. W. H. Taylor, Cavendish Laboratory, Cambridge, England.

BOOK REVIEWS

PETROCHEMISCHE BERECHNUNGS METHODEN AUF ÄQUIVALENTER GRUNDLAGE, by CONRAD BURRI. 334 pages, 66 figures. Birkhäuser Verlag, Basel and Stuttgart, 1959. Price not indicated.

This book is the work of two men, one the author, Professor Conrad Burri, Professor of the Eidgenössischen Technischen Hochschule and the University of Zurich, and the other, the late Professor Paul Niggli, who devised the techniques of petrochemical presentation that are described in the book.

The book has five parts: A) Introduction, B) Petrochemical calculation methods on equivalent basis, as devised by Paul Niggli, C) The proposals of Barth and Eskola toward considerations of the anions in petrochemical calculations and their relationships to the equivalent norm of Niggli, D) Appendix, E) References. This is a book which has long been necessary and for which there should also be an English equivalent. One of the few articles in English readily available to American petrologists which describes Niggli's calculations is that by C. S. Bacon, Jr., published in the *American Mineralogist*, **32,** 257–295, 1947. There is also information on the so-called Niggli values in Parker's English version of Volume 1 of Niggli's projected three-volume work, "Rocks and Mineral Deposits" (*Am. Mineral.*, **34,** 128–129, 1949; **40,** 131–132, 1955). The major part of the book, approximately 210 pages, is devoted to part B, which itself is split into two sections, 1) the Niggli values si, al, fm, c, alk, k, mg, ti, p, etc., and their application, and 2) the equivalent norm. The presentation is everywhere clear, and numerous examples are given to illustrate the calculations.

Part C, dealing with Barth's and Eskola's extensions of the method, includes not only the calculation of rock analyses with reference to the additional anions, but also describes Barth's standard cell. References in English to this procedure are in Barth's book, "Theoretical Petrology," pp. 82–85, which also contains a discussion of the Niggli values, their calculation and application (*Am. Mineral.* **37,** 1067–1068, 1952).

The Appendix contains three tables: 1) an alphabetical list of the base and equivalent normative compounds, 2) a listing of the important equations of reactions between base and equivalent normative compounds, and 3) tables for calculating molecular and atomic equivalent numbers for the most important rock-forming oxides. The book concludes with a list of the significant literature, and author and subject indexes.

It is unfortunate that this method of presentation has received relatively little detailed attention by American petrologists. Doubtless this has been due in part to the fact that most of the significant literature has been in German, much of it under Niggli's authorship. Geology students beginning their study of German have generally shied away from Niggli's works owing to the abstruse nature of his grammatical presentations. It is to be hoped that this general summary by Burri will greatly expand the interest of English-speaking students in this important petrochemical technique.

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IGNEOUS AND METAMORPHIC PETROLOGY, Second Edition by FRANCIS J. TURNER AND JOHN VERHOOGEN. 694 pages. McGraw Hill Book Company, Inc., New York, N. Y. $12.00.

The First Edition of "Igneous and Metamorphic Petrology" by Turner and Verhoogen was reviewed in the *American Mineralogist*, Vol. 37, pp. 702–703, 1952. As was anticipated at that time, the book has met with a highly favorable reception from American students of petrology; indeed, without doubt it has become the foremost modern summary of principles, problems, and theories in igneous and metamorphic petrogenesis.

Much of the Second Edition has been largely rewritten in order to include many of the more recent results in experimental petrology and geochemistry. The book is approximately 90 pages longer than the First Edition. The chief expansion has taken place in Chapter 5, entitled "Crystallization of Igneous Minerals from Silicate Melts." One new chapter has been added (Chapter 13) on nepheline syenites, ijolites, and carbonatites; and pegmatites, Chapter 14, have been placed in a separate chapter.

The presentation of material in the second half of the book, on metamorphism, has been completely rearranged, and Chapters 18–24 of this part have been revised, largely based on Memoir 73 of the Geological Society of America—"Metamorphic Reactions and Metamorphic Facies" by W. F. Fyfe, F. J. Turner, and J. Verhoogen, 1958. Thus this edition contains two more chapters than the first.

All of these changes contribute significantly to the improvement of the book and assure its continuing popularity. As evidence of its general acceptance, the book has now been placed by the publishers in their International Series in the Earth Sciences.

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GENERAL CRYSTALLOGRAPHY, a Brief Compendium, by W. F. deJong with the collaboration of J. Bouman. W. H. Freeman and Company, San Francisco, California, 1959. 281 pages, 231 figures, 41 tables. Price $6.00.

This book is not intended for a textbook but is an excellent summary of crytallography, clearly written and well illustrated. Part I, Geometrical crystallography (92 pages), covers crystal description, calculation and drawing. Part II, Structural crystallography (42 pages) includes experimental methods, Fourier analysis and Patterson diagrams. Part III, Chemical crystallography (53 pages) discusses crystal bonds and structure types. Part IV, Physical crystallography (85 pages) covers homogeneous deformation, stress, the crystal as a dielectric, thermal and electronic conduction, pyro- and piezoelectricity, elasticity, imperfections, gliding, and crystal growth. There are numerous references, and both a subject and author index. This is a slightly enlarged version of deJong's Kompendium der Kristallkunde, published by Springer-Verlag, Vienna, in 1959.

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CRYSTALS AND CRYSTAL GROWING, by Alan Holden and Phylis Singer. Science Study Series—Anchor Books, Doubleday and Co., Inc., Garden City, N. Y., 1960. 320 pages, 137 figures, 42 plates, 4 color inserts. Price $1.45 ($1.65 in Canada).

This is a good answer to the question "What book about crystals can I give to a high school student?" It should even be interesting to an adult. It contains a surprising amount of information concerning crystals, clearly written in simple language. It discusses such subjects as solutions, crystal growth, symmetry, habit, cleavage, arrangement of atoms in solids, and methods for growing crystals. There are numerous examples and illustrations. Questions concerning crystals and suggested simple research problems are included.

There are some minor points, especially of emphasis, which might be questioned, but the reviewer is more inclined to praise the authors for presenting a difficult subject in such an interesting and intelligible manner.

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PROCEEDINGS OF THE SECOND UNITED NATIONS INTERNATIONAL CONFERENCE ON THE PEACEFUL USES OF ATOMIC ENERGY—VOLUME III, SURVEY OF RAW MATERIAL RESOURCES, UNITED NATIONS PUBLICATION, 1958, 845 pages, International Documents Service, Columbia University Press, New York. $18.50.

The first book (Volume 6) on geology of uranium and thorium issued following the first general conference on peaceful uses of atomic energy (1955) consisted of the reports of two sessions, 6B and 7B. Most of the articles (Session 6B) dealt with geology and mineralogy of uranium and thorium deposits. Thirty-one reports described deposits outside of the United States and 69 gave information on areas and deposits in the United States. The 27 papers in session 7B dealt with prospecting methods for radioactive ores in various parts of the world.

The new volume of the complete series of 33 on the Proceedings of the second International Conference on the Peaceful Uses of Atomic Energy (1958) contains the results of three sessions—E 5, E-7, and E-9 under the title "Survey of Raw Materials Resources." In the three years since the appearance of the geology-mineralogy volume of the first congress, many new discoveries and techniques in the search for uranium have been made and developed. More emphasis has been placed on other types of nuclear raw materials, namely beryllium, zirconium, and thorium. There also have been satellite studies in uranium geochemistry and a detailed study of uranium mineralogy. This volume also is noteworthy for the increase in participating investigators from countries outside of the United States.

The book is subdivided into Raw Materials Supply (E-5, 118 pages) consisting of 17 papers; Geochemistry (E-7, 153 pages), with subdivision (A) Geochemical Prospecting comprising 15 articles, and (B) Isotopic Composition and Age Determination consisting of 9 articles. This is followed by the greatest number of papers in Session E-9 (563 pages). This last session, entitled Mineralogy and Geology and Prospecting is subdivided into (A) Mineralogy and Genesis of Deposits (21 articles), (B) Geology of Deposits (30 articles), and (C) Prospecting (10 articles).

Opening topics on Resources of Nuclear Fuel for Atomic Power, Geologic Distribution of Nuclear Raw Materials and Geologic Appraisal of Uranium Resources of the United States are followed by articles on zirconium and beryllium resources. Next come discussions on uranium, thorium, and other reactor materials in Canada, France, Union of South Africa, Egypt, India, and Japan.

Session E-7, Geochemistry, includes information on new techniques in uranium exploration such as geochemical methods, chemical prospecting, a study of uranium in groundwater, springs, humus, petroliferous rocks, and the use of such tools as electron microscope and paper chromatography. This section is followed by papers on isotopic lead studies, age measurements, radioactive disequilibrium and beryllium geochemistry. A concluding paper deals with isotopic studies on meteorites and the Earth's crust.

Session E-9 begins with a summary of recent progress in the mineralogy of uranium, followed by new data on uranium minerals of the USSR. Subsequent miscellaneous articles discuss uranium migration, ore genesis, and origin of certain deposits. Particularly noteworthy is an article by Liebenberg on the controversial Witwatersrand ores. Other topics are the occurrence of uranium in coals, shales, hydrothermal ores, and the paragenesis of USSR deposits. Under geology of the deposits are descriptions of Canadian ores bodies, a few articles on various deposits of the United States, Argentina, Greenland, France and French Union, Portugal, Belgium Congo, India, and Japan. Lastly, there are discussions of prospecting methods and various radioactive measurements.

In general the illustrations are clear, but reduction of some maps has produced some illegible results, which was also the case in the earlier volume.

To the reviewer the increase in the number of articles from investigators outside of the United States greatly enhances the value of the book, particularly as these present new ideas that may be applied to problems on the geology and mineralogy of domestic deposits. However, it is disappointing to note that all of the articles submitted by the USSR geologist leave out locality names—a unfortunate omission. The book is well worth adding to geology libraries, but its high cost (considerably higher than the 1955 volume) limits its acquisition by individual geologists.

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STRUCTURE REPORTS FOR 1952. Vol. 16. General Editor, A. J. C. Wilson, Asst. Editor, Dubravka Havranek. Section Editors: N. C. Baenziger, Metals; J. Wyart, Inorganic Compounds; J. Monteath Robertson, Organic Compounds. Published for the International Union of Crystallography by N. V. A. Oosthoek's Uitgevers Mij, Utrecht, Netherlands. Price $32.00; £11.6.0.

The excellent quality of abstracts and the same type of arrangement found in previous volumes are continued in this volume covering the crystal structure literature of 1952.

STRUCTURE REPORTS, Vol. 14. Supplementary volume, and cumulative index for 1940–1950. General Editor, A. J. C. Wilson, Asst. Editor, O. Eisner. Section Editors: N. C. Baenziger and C. S. Barrett, Metals; J. M. Bijvoet and J. Wyart, Inorganic Compounds; J. Monteath Robertson, Organic Compounds. Published for the International Union of Crystallography by N. V. A. Oosthoek's Uitgevers Mij, Utrecht, Netherlands. Price $9.50; £3.80.

Previously unreported abstracts, together with cumulative subject and author indices, and cumulative corrigenda are included.

NEW MINERAL NAMES

Cafetite

A. A. Kukharenko, V. V. Kondrat' Eva, and V. M. Kovyazina. Cafetite, a new hydrous titanate of calcium and iron. *Zapiski Vses. Mineralog. Obshch.*, **88,** No. 4, 444–453 (1959) (in Russian).

Analysis of the mineral gave TiO_2 54.11, Al_2O_3 2.02, Fe_2O_3 22.00, FeO 0.49, MgO 1.44, MnO 0.20, CaO 6.23, Na_2O 0.28, K_2O 0.40, H_2O^+ 8.83, H_2O 3.46, sum 100.46%, corresponding to (Ca, Mg) $(Fe, Al)_2$ Ti_4O_{12} $4H_2O$. Spectrographic analysis showed also V~0.03, Zr 0.001–0.003, Nb 0.003–0.01, and Y~0.1%. A dehydration curve shows that about one-fourth of the water is lost below 120°, about one-half at 200–300° and the remainder up to 650°. Insoluble in HCl and HNO_3, decomposed by boiling concentrated H_2SO_4. Fusible in the reducing flame to a dark brown, weakly magnetic mass.

Crystals of cafetite are columnar to acicular, elongated along (001), and are striated on the prism faces. The dominant forms are {100}, {520}, and {210}, also noted {320}, {310}, {410}, {610}, {810}, {052}, and {801}. Goniometric data are given; the measurements were not very satisfactory.

Laue and rotation photographs indicated orthorhombic symmetry, (but the optics disagree), with *a* 31.34, *b* 12.12±0.03, *c* 4.96±0.01 kX, $a:b:c$ 2.5858:1:0.4092, $Z=6$. Unindexed *x*-ray powder data are given (25 lines); the strongest lines are 7.84 (10), 3.26 (8), 2.557 (8), 1.910 (7). Cleavage prismatic, in two directions.

The mineral is pale yellow to colorless, luster adamantine. Hardness 4–5, brittle, but needles show elasticity. G. 3.28 (measured), 3.19 (calcd). Optically biaxial, negative, with *ns*, α 1.95, β 2.08, γ 2.11. $Z:c=2$–4°, hence the mineral is monoclinic. Dispersion strong.

The mineral is one of the last minerals to form in miarolitic cavities in pegmatites cutting the Afrikanda pyroxenite massif, Kola Peninsula, U.S.S.R. Associated minerals include titanomagnetitite, phlogopite, ilmenite, sphene, clinochlore, dysanalyte, baddeleyite, anatase, and an unidentified mineral (see page 479). Cafetite forms tangled fibrous aggregates and radial-fibrous aggregates of acicular crystals.

The name is for the chemical composition.

Michael Fleischer

Calciotalc

D. P. Serdyuchenko. Calciotalc, a new mineral. *Zapiski Vses. Mineralog. Obshch.*, **88,** 298–304 (1959) (in Russian).

N. V. Belov. On the calciotalc of D. P. Serdyuchenko. *Ibid.*, 305–30i (1959) (in Russian).

Hydrothermal alteration of diopside actinolite of Archean rocks in the basin of the Leglier River, Aldan Region, southern Yakutia, has caused the formation of a peculiar talc (calciotalc) as partial or complete pseudomorphs after actinolite. The contact with actinolite is sharp. Also present in greater abundance is normal talc; the transition of calciotalc into the normal talc is gradual.

Analyses by V. A. Yusova are given of the actinolite, calciotalc, and talc.

The analysis of calciotalc corresponds to the formula

$$(Ca_{0.79}R'_{0.03}Mg_{1.96}Fe''_{0.12}R'''_{0.06})(Si_4O_{10})[(OH)_{1.50}O_{0.24}].$$

The mineral has columnar structure (preserved from the actinolite), is dense and comparatively hard (between 2 and 3). It is greenish-gray with silky to pearly weak luster. In thin section it is colorless (the actinolite is colored and pleochroic), optically biaxial, negative, 2 V small, extinction parallel on Z, *ns* α 1.565, γ1.583, both ±0.001.

	1 *Actinolite*	*2* *Calciotalc*	*3* *Talc*
SiO_2	50.94	60.94	58.68
TiO_2	0.16	Trace	0.04
Al_2O_3	4.82	0.23	1.15
Fe_2O_3	1.40	0.80	1.23
FeO	7.30	2.16	3.85
MnO	0.11	0.04	0.01
MgO	18.65	20.00	27.26
CaO	12.71	11.18	1.43
Na_2O	0.58	0.29	0.18
K_2O	0.80		
CO_2	None	None	None
F	1.18	—	—
Cl	0.16	—	—
H_2O^+	0.91	3.40	5.10
H_2O^-	0.24	0.98	1.11
BaO	0.04	—	—
Li_2O	0.05	—	—
	100.05	100.02	100.04
$-(O=F_2Cl_2)$	0.53		
	99.52		

The *x*-ray powder diagram by V. G. Muraveiska is similar to that of talc, but with larger spacings; the strongest lines are 9.25 (9), 3.079 (9), 1.518 (6), 2.469 (5b).

Belov compares the compositions and structures of the minerals:

Talc	Mg_3	$Si_4O_{10}(OH)_2$
Calciotalc	$CaMg_2$	$Si_4O_{10}(OH)_2$
Margarite	$CaAl_2$	$(Si_2Al_2)O_{10}(OH)_2$,

and argues that calciotalc is related more closely to margarite than to talc, as shown especially by the hardness. He points out that the name may therefore not be a suitable one.

Discussion.—Detailed structural analysis of calciotalc and margarite would be highly desirable.

M. F.

Ikunolite

Akire Kato. Ikunolite, a new bismuth mineral from the Ikuno Mine, Japan. *Mineralogical Journal (Japan)*, **2**, No. 6, 397–407 (1959).

Analysis by T. Katsura on a small amount of hand-picked material gave Bi 79.69, S 8.89, Se 1.98, Rem. (=ferberite and quartz) [9.44], sum [100.00%]. Minor amounts of Ag and Te (probably less than 0.1%) were detected by *x*-ray spectrography. The analysis gives Bi:S:Se—3.79:2.76:0.24 or $Bi_4(S, Se)_3$.

Laue and oscillation photographs show ikunolite to be rhombohedral, space group $D^5_{3d}-R\bar{3}m$, $a_{rh}=13.28$ Å, α 18°00′, Z=1; hexagonal lattice, *a* 4.15, *c* 39.19 Å, Z=3. Cleavage basal, perfect. Indexed *x*-ray powder data are given; the strongest lines are 3.022 (100), 2.205 (30), 6.56 (20), 3.536 (20), 2.076 (20), 1.865 (20). The structure is worked out in detail.

The mineral is lead gray, streak dark gray, luster metallic. G. 7.8 (measured), 7.97 (cold), H. 2, very flexible. In polished section white with faintly creamy tint, nearly isotropic in basal section, moderately anisotropic pale gray to gray in diagonal section. Etch tests are given.

The mineral occurs in a quartz vein from the Ikuno Mine, Hyôgo Prefecture, Japan, associated with ferberite, bismuth, and bitmuthinite, with some cassiterite.

It is very close in crystallographic and physical properties to joseite A (Thompson, *Am. Mineral.* **34,** 342–382 (1949)); it is to be classed with the tetradymite group.

The name is for the mine.

M. F.

Strontioginorite

Otto Braitsch.—Über Strontioginorit, ein neue Ginorit—Varietät aus dem Zechsteinsalz. *Beitr. Mineral. u. Petrog.*, **6,** 366–370 (1959).

The mineral was found in the water-insoluble residues of the "Old Halite" bed (NaCl about 96%, anhydrite 2–4%) from the Königshall-Hindenburg potassium mine at Reyershausen, northern Germany. It forms crystals up to 3 mm. long, 1 mm. wide, and 0.5 mm. thick, but most are much smaller.

There was insufficient material for analysis. Spectrographic study showed B, Sr, and Ca major; *x*-ray fluorescence analysis indicated SrO 10–20%. Assuming the formula $(Sr, Ca)_2B_{14}O_{23}\cdot 8H_2O$, the molecular weight calculated from the unit cell and specific gravity gave Sr:Ca about 1.3:0.7.

The crystals are tabular, dominant forms are (110), (010), and (111); also noted were (001), (100), (250), (131), (141), and doubtfully (151). Rotation and Weissenberg photographs gave: Monoclinic, space groups $P2_1/a - C^5_{2h}$, *a* 12.85, *b* 14.48, c12.84$_5$, all ±0.01 Å, β 101°35′±10′, Z=4. Cleavage (010) perfect and easy, (001) very good. The crystals are easily deformed. Indexed *x*-ray powder data are given; the strongest lines are 7.25 vs, 2.10 s, 5.40 m, 3.92 m, 3.34 m, 1.19 m.

The mineral is colorless, transparent, with silky luster. Hardness 2–3, G. (suspension) 2.25. Optically bixial, positive, *ns* (Na), α 1.512, β 1.524, γ 1.577, all ±0.003, 2V 53°, $Y=b$.

Braitsch points out that the unit cell and indices of refraction are very close to those given for the KSr borate volkovite, see *Am. Mineral.*, **40,** 551–552 (1955).

Discussion.—If Sr > Ca, this is not a variety of ginorite, but its strontium analogue.

M. F.

Sulunite

A. A. Nyrkov. Sulunite a new mineral of the ferriferous chlorites. *Zapiski Vses. Mineralog. Obshch.*, **88,** 571–577 (1959) (in Russian).

Analysis by P. P. Ovcharenko gave SiO_2 40.31, Al_2O_3 28.69, total iron as Fe_2O_3 12.82, MgO 0.52 CaO 0.15, K_2O 4.00, Na_2O 3.16, SO_3 0.77, loss on ignition 7.82, sum 98.24%. Qualitative tests showed FeO to be present; an exothermal break at 460° with a gain in weight is interpreted as showing that approximately all the iron is ferrous. The analysis should also be corrected for 0.27% calcite, 1% carbonaceous matter, and 1.34% pyrite. A dynamic loss of weight curve shows losses in weight: 0–200° 1.86%, 200–350° 0.83%, 350–450° 0.83%, 450–480°-1.10% (gain) 480–720° 7.18%, 720–900° 0, 900–950° 0.55%. A D.T.A. curve showed endothermal breaks at 50–200°, 600°, and 930° (calcite), exothermal breaks at 300° and 460°.

The mineral is greenish, fibrous, soft and greasy to the touch. G. 2.95. One good cleavage. Optically biaxial, negative, *ns* α 1.574, β 1.595, γ 1.598, 2V 42°, extinction along fiber parallel, elongation positive. Absorption X > Y > Z, dispersion weak.

X-ray powder data (27 lines) are given; the strongest lines are 7.08 (10), 13.4 (7, broad), 4.68 (7), 3.313 (6), 2.795 (6), 1.530 (6), 7.35 (5), 7.14 (5), 3.51 (5), 1.493 (5). The pattern resembles those of sheridanite and leuchtenbergite, given for comparison.

The mineral occurs in shales and sandstones as thin incrustations on fossil plants; it is always associated with carbonaceous matter. Found in a number of drill cores from the Sulinsk region, Donetz Basin, U.S.S.R.

It is similar to the chlorites in x-ray pattern, but differs in the low Mg and high alkali content, and in having high birefringence. It differs chemically and in x-ray pattern from gümbelite.

The name is for the Sulinsk region.

Discussion.—Needs further study. The analysis is unsatisfactory; both FeO and Fe_2O_3 are likely to be present and the summation is very low. Must be proved not to be a mixture.

M. F.

Yoshimuraite

Takeo Watanabe. The minerals of the Noda-Tamagawa Mine, Iwate Prefecture, Japan. I. Notes on geology and paragenesis of minerals. *Mineralogical Journal* (*Japan*), **2,** 408–421 (1959).

The name and formula $(Ba, Sr)_4 Mn_4 Ti_2 (SiO_4)_4 [(P, S)O_4]_2 (OH, Cl)_3$ are given for a mineral occurring in peculiar pegmatitic rocks on the foot-wall side of the ore zone. Associated minerals are barium-bearing feldspar, rhodonite, urbanite, and richterite. No further data are given; the paper describes the paragenetic relations in a very unusual deposit (50,000 tons of pyrochroite-manganosite ores have been mined).

M. F.

Unnamed

A. A. Kukharenko, V. V. Kondrat' Eva, and V. M. Kovyazina. Cafetite, a new hydrous titanate of calcium and iron. *Zapishi Vses. Mineralog. Obshch.*, **88,** No. 4, 444–453 (1959) (in Russian.)

Associated with cafetite (see page 476) is an unknown mineral, which occurs as pseudomorphs after perovskite and dysanalyte, replaces ilmenite, and forms platy overgrowths on titanomagnetite. It is pale yellow, luster adamantine, has one perfect cleavage. H about 5, G. 3.40–3.45. Under the microscope it is pale yellow and shows polysynthetic twinning. Optically biaxial, negative, 2V 35–40°, dispersion $r>v$, very strong. The extinction is oblique to the twinning edge. *ns* α about 1.950, β 2.13, γ about 2.24. Spectrographic analysis showed Ti and Ca major, also Nb, Ce, Y, Fe. The mineral is insoluble in acids; loss of weight 8.5% when heated. X-ray powder data (37 lines) are given; the strongest lines are 3.30 (10), 1.761 (10), 4.77 (5), 3.63 (4), 2.29 (4), 1.501 (4).

M. F.

NEW DATA

Arsenolamprite

Zdenek Johan. Arsenolamprit—die rhombische Modifikation des Arsens aus Černy Dul (Schwarzenthal) im Riesengebirge. *Chem. Erde*, **20,** 71–80 (1959).

Arsenolamprite, the supposed dimorph of arsenic, was recently discredited (*Am. Mineral.* **43,** 1225 (1958)). It is now re-established from a new locality, Černy Dul, where it occurs as dark gray plates and veinlets in carbonate. Spectrographic analysis showed As major, Fe, Ca, Mg, Al, Si minor, Ag, Sb small, Be, Bi, Cr, Cu, Hg, Mn, Pb, Ti, Zn traces. The Ca, Mg, Al, Si are attributed to admixed carbonate, the Ag and Hg to native silver, the Fe in part to loellingite and a chlorite-like mineral. In reflected light the mineral is gray-white, very weakly anisotropic, reflecting power close to that of rhombohedral As. Etch tests (1 minute): $FeCl_3$, 20%, neg. (distinction from arsenic), HCl, 1–1, $HgCl_2$, 5%, and KOH, 40%, all negative; HNO_3, 1–1, positive, but much less attacked than arsenic. The *x* ray powder photograph resembles that of black phosphorus and is indexed on that basis, giving an orthorhombic cell with *a* 3.63, *b* 4.45, *c* 10.96 Å, Z=8, G calcd. 5.577. The strongest lines are 5.44 (vs), 2.720 (vs), 1.115 (vs), 2.740 (s), 1.731 (s), 3.48 (m), 2.235 (m), 1.877 (m), 1.815 (m), 1.520 (m), 1.362 (m), 1.261 (m), 1.209 (m).

M. F.

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Hoppin, Richard A.
Luth, William C.
Runner, J. J.
Sargent, Kenneth A.
Orange City
Boone, Jon A.

KANSAS

El Dorado
Knupp, Richard C.
Lawrence
Franks, Paul C.
Gillerman, Elliot
Pooser, William K.
Swineford, Ada
Manhattan
Lobmeyer, David H.
Riseman, Louis
Sperry, Arthur B.
Wingard, Paul S.
Topeka
Barkes, William R.
Rodekohr, W. J.

KENTUCKY

Lexington
Brown, William R.
Fisher, Irving S.
Louisville
Johnson, David M.
Neiman, Robert

LOUISIANA

Baton Rouge
Winner, Maurice D., Jr.
Ruston
Colombini, Victor
Shreveport
Rosenkrans, R. R.
Trichel, Hall

MAINE

Auburn
Woodman, Raymond G., Jr.
Brunswick
Bodine, Marc W., Jr.
Orono
Osberg, Philip H.
Trefethen, Joseph M.
Roxbury
McCrillis, Dean A.

MARYLAND

Baltimore
Cohan, Samuel H.
Donnay, Gabrielle
Donnay, J. D. H.
Wise, William S.
Bel Air
Rushton, Edward W., II
Beltsville
Albee, Arden L.
Heyl, Allen V., Jr.
Mertie, John B., Jr.
Overstreet, Elizabeth F.
Pavlides, Louis
Bethesda
Clark, Joan R.
Greenwood, Hugh J.
Larsen, Esper S., III
Ordway, Fred
Brandywine
Laughlin, A. W.
Catonsville
Wieczorek, George A.
Chevy Chase
Magin, George B., Jr.
White, Donald E.

- White, Walter S.

Chillum
- Smith, William R.

College Park
- Ampian, Sarkis G.
- Bourbeau, Gerard A.

Hyattsville
- Parker, John G.

Kensington
- Pommer, Alfred M.
- Postel, A. Williams
- Stieff, Lorin R.

Landover
- Dwornik, Edward J.

Mt. Airy
- Darneal, Robert L.

Port Republic
- Karrer, Sebastian

Rockville
- Davis, Gordon L.

Silver Spring
- Adler, Hans H.
- Ashby, George E.
- Cuttitta, Frank
- Faust, George T.
- Hanley, John B.
- Hendricks, S. B.
- Hill, William L.
- Hooker, Marjorie
- Johnson, Serah C.
- MacDonald, Gordon J. F.
- Meyrowitz, Robert
- Ramisch, J. L.
- Robertson, Eugene C.
- Robie, Richard A.
- Roth, Robert S.
- Smith, Deane K.
- Zen, E-an

Takoma Park
- Feinstein, Hyman I.

Towson
- Brown, John S.

Washington Grove
- Boardman, Robert L.

MASSACHUSETTS

Amherst
- Bain, George W.
- Brophy, Gerald P.
- Johnson, Steven B.
- Moores, John C., Jr.
- Nelson, Albert B.

Arlington
- Burnham, Charles W.

Bedford
- Yagoda, Herman

Belmont
- Frondel, Judith W.
- Stanwood, Creighton B.

Boston
- Bjareby, Gunnar
- Gheith, M. A.
- Wolfe, C. W.

Braintree
- Enzmann, Robert D.
- Wolfson, Leila
- Wolfson, Sumner H.

Brockton
- DiCroce, Robert N.

Brookfield
- Coes, Loring, Jr.

Brookline
- Lancaster, Robert A.
- Wall, Milford W.

Cambridge
- Aristarain, Lorenzo F.
- Bilgrami, S. A.
- Billings, Marland P.
- Boucot, Arthur J.
- Buerger, Martin J.
- Carpenter, Alden B.
- Dixon, H. Roberta
- Fairbairn, H. W.
- Frondel, Clifford
- Garrels, Robert M.
- Gaudin, A. M.
- Hatch, Norman L., Jr.
- Hostetler, Paul B.
- Hurlbut, Cornelius S.
- Ito, Jun
- McKinstry, H. E.
- Mencher, Ely
- Michelson, Robert A.
- Milton, Daniel J.
- Moore, John M., Jr.
- Navias, Robert A.
- Perry, Eugene C., Jr.
- Powell, James L.
- Prewitt, Charles T.
- Ross, Daphne R.
- Ross, Malcolm
- Sato, Motoaki
- Simmons, Marvin G.
- Thompson, James B., Jr.
- Thompson, Mary E.
- Truesdell, Alfred H.
- Washken, Edward
- West, C. D.
- Wuensch, Bernhardt J.

Easthampton
- Schortmann, Raymond E.

Hudson
- Guidotti, Charles V.

Ipswich
- Robertson, Elizabeth J.

Lexington
- Davis, Carlton M.

Maynard
- Linial, Andrew V.

Melrose
- Furlong, Ira E.

Needham
- Jarrell, Oscar W.

Needham Heights
- Croft, William J.

Newton
- Ladoo, Raymond B.

Newton Highlands
- Dearborn, Grace G.

Newton Lower Falls
- Draisin, Wilburt M.

Northampton
- Shaub, B. M.
- Stobbe, Helen R.

Northborough
- Hight, Richard P.

Pittsfield
- Kernaghan, James S.

Revere
- Mallette, Reese E., Jr.

Roxbury
- Vilks, Irita

Somerville
- Mariano, Anthony N.

South Egremont
- Besson, William W.

South Hadley
- Haff, John C.

Stoneham
- Zibell, Stuart

Walpole
- Ruggles, Richard D.

- Watertown
  - Hunt, Mirla E.
- West Peabody
  - Krueger, Harold W.
- Williamstown
  - Perry, Elwyn L.
- Winchester
  - Brown, Nelson K.
  - Giacalone, Joseph J.
  - MacKenzie, Kenneth L.
- Woburn
  - Pitha, Carl A.
- Worcester
  - Sand, L. B.
  - Sonneborn, John P., Jr.
  - Thibault, N. W.

MICHIGAN

- Ann Arbor
  - Archbold, Norbert L.
  - Blair, Robert G.
  - Chase, George A.
  - Conrad, Malcolm A.
  - Cooley, Mary E.
  - Denning, Reynolds M.
  - Fear, Robert H.
  - Gardner, Bernard G.
  - Goddard, E. N.
  - Gross, Eugene B.
  - Heinrich, E. Wm.
  - Hunt, Walter F.
  - Kneller, William A.
  - Krause, Edward H.
  - Landes, Kenneth K.
  - Perhac, Ralph M.
  - Quashie, Lloyd A. K.
  - Ramsdell, Lesis S.
  - Shappirio, Joel
  - Slawson, Chester B.
  - Turneaure, F. S.
  - Weiss, Benjamin
- Chassell
  - Ludeman, Frank L.
- Detroit
  - Insley, Robert H.
  - Koebel, Charles J.
  - Parsons, Willard H.
  - Williams, Edward E.
- East Lansing
  - Cummings, David
- Flint
  - Barlett, Helen Blair
- Holly
  - Schwartzwalder, Karl
- Houghton
  - Doane, Mrs. Virginia L.
  - Sigsby, Robert J.
  - Snelgrove, A. K.
  - Spiroff, Kiril
  - Wotorson, Cletus S.
- Iron River
  - Zimmer, Paul W.
- Ishpeming
  - Han, Tsu-Ming
- Kalamazoo
  - Garske, David H.
- Midland
  - Rinn, Harold W.
- Royal Oak
  - Barczak, Virgil J.
- Tecumseh
  - Handy, Arlene M.
- Whitmore Lake
  - Ryan, Richard C.

MINNESOTA

- Duluth
  - Green, John C.
  - Lepp, Henry
- Hibbing
  - Scott, Donald W.
- Minneapolis
  - Bastien, Thomas W.
  - Blake, Roland L.
  - Cooke, S. R. B.
  - Ellestad, R. B.
  - Gast, Paul W.
  - Gruner, John W.
  - Gundersen, James N.
  - Holler, Albert C.
  - Muncaster, Neil N.
  - Phinney, William C.
  - Sandell, E. B.
  - Schultz, Gerald E.
  - Schwartz, G. M.
  - Zoltai, Tibor
- Nashwauk
  - Beckmann, Charles A.
- Northfield
  - Hagen, Norland K.
  - Stewart, Duncan
- Red Wing
  - Chesley, Frank G.
- Rochester
  - Kennedy, Fred C.
- St. Paul
  - Bayer, Thomas N.
  - Boerbon, Floyd W.
  - Cadwell, Donald E.
  - Papike, James J.
  - Thill, Richard E.
  - Titcomb, Jane
  - Wewerka, Eugene M.
- White Bear Lake
  - Hatch, Robert A.

MISSISSIPPI

- Hattiesburg
  - Harding, James L.
- Jackson
  - Buck, Alan D.
  - Johnson, Wendell B.
  - Mather, Mrs. Bryant

MISSOURI

- Clayton
  - Moffat, James D., III
- Columbia
  - Hubert, John F.
  - Johnson, Clayton H.
  - Keller, W. D.
  - Marshall, C. Edmund
  - Montgomery, Joel K.
  - Sleeman, Lyle H., Jr.
  - Toler, Larry G.
- Ironton
  - Morrison, Bert C.
- Jennings
  - Stueber, Alan M.
- Joplin
  - Ritchie, Everett J.
- Mexico
  - Green, Robert S.
  - Ponder, Herman
- Rivermines
  - Brophy, Rosser C.
- Rolla
  - Amstutz, G. C.
  - Chan, Samuel S. M.
  - Grawe, O. R.
  - Kennedy, Richard R.

St. Louis
- Allen, Victor T.
- Hurst, Thomas L.
- Nason, Howard K.
- Spence, Wayne G.
- Tolman, Carl
- Weeks, Wilford F.

Salem
- Kisvarsanyi, Eva B.

MONTANA

Anaconda
- Dahlem, David H.
- Dudas, Laszlo

Bozeman
- Foster, Robert J.
- Tompson, Willard D.

Butte
- Crowley, Frank A.
- Grunig, James K.
- Zeihen, Lester G.

Corvallis
- Erickson, Louis

Great Falls
- Erdmann, Charles E.

Missoula
- Hood, William C.
- Hower, John
- Wehrenberg, John P.

Wibaux
- Johnson, Hjalmar

NEBRASKA

Omaha
- Bennett, Mordecai
- Hayes, John B.
- Heck, William J.

South Sioux City
- Allen, James

NEVADA

East Ely
- Berge, Byron T.

Las Vegas
- Nelson, Harry E.

Reno
- Cooke, H. R., Jr.
- Gianella, Vincent P.
- Lawrence, Edmond F.
- McGirk, Lon S., Jr.
- Palmer, James B.
- Scheid, Vernon E.
- Stathis, George J.
- von Volborth, Alexis

NEW HAMPSHIRE

Colebrook
- Newman, William A.

Dover
- Foster, Philip C.

Durham
- Atherton, John S.
- Meyers, T. R.
- Schneer, Cecil J.
- Stewart, Glenn W.

Exeter
- Burbank, Wilbur S.

Hanover
- Barnard, Walther M.
- Page, Lincoln R.
- Page, Norman J.
- Stoiber, Richard E.

Hillsboro
- Richmond, W. E.

West Lebanon
- Lyons, John B.

NEW JERSEY

Belmar
- Townes, William D.

Bergenfield
- Smith, Arthur E., Jr.

Blairstown
- Cook, Mrs. E. Packard

Bloomfield
- Luedemann, Lois W.

Boonton
- Hendricks, John G.

Bound Brook
- Cummins, A. B.

Chester
- Kadey, Frederic L., Jr.

Clifton
- Mullner, Henry C.

Dover
- Allen, Walter C.

Edison
- Croad, George F.

Elberon
- Tauber, Arthur

Elizabeth
- Bundy, Wayne M.
- Murray, Haydn H.
- Swayze, Donald R.

Fair Haven
- Nye, William F.

Fanwood
- Humphrey, Marshall F.
- Partridge, Padraic

Flemington
- Comeforo, J. E.

Fort Lee
- Bollin, Edgar M.

Fort Monmouth
- Kohn, J. A.
- Levin, S. Benedict

Franklin
- Baum, John L.
- Callahan, William H.

Glen Ridge
- Taylor, James A.

Haddonfield
- Morgan, Leonard A.

Iselin
- Servilla, Thomas

Lake Hiawatha
- Klein, Matthew

Leonia
- Aldrich, Henry R.

Lincroft
- Toman, L., Jr.

Linwood
- Kelly, Joseph C.

Little Silver
- Kedesdy, H. H.
- Wolff, G. A.

Manville
- Harker, Robert I.
- Meyer, Jurg W.
- Pundsack, Fred L.
- Yang, Julie C.

Menlo Park
- Oulton, T. Dixon

Metuchen
- Howard, Nancy W.

Millburn
- Gosling, Beatrice M.

Mountain Lakes
- Wilcox, John T.

Mountainside
- Wintringham, Neil A.

Murray Hill
- Geller, Seymour

Laudise, Robert A.
Wood, Elizabeth A.
Newark
Buckelew, Harold C.
New Brunswick
Douglas, Lowell A.
Lodding, William
Martens, J. H. C.
Wilkerson, Albert S.
North Plainfield
Barr, Henry B.
Mushlitz, Richard D.
Orange
Fleischer, Arthur
Plainfield
Jessen, Heinrich C.
Princeton
Aleshin, Eugene
Buddington, Arthur F.
Hess, Harry H.
Menendez, Alfredo
Nagle, Frederick, Jr.
Nolf, Bruce O.
Sampson, Edward
Ridgewood
Mitchell, H. Allan
Scotch Plains
Sabo, Joseph R.
Skillman
Knapp, Guenever P.
Sparta
Edwards, Frank Z.
Stirling
DuPont, James M.
Summit
Howard, Calhoun L. H.
Lamb, Harold A.
Trenton
Di Piazza, James J.
Union
Albanese, John S.
West Caldwell
Sachs, Walter P.
West End
Giardini, A. A.
Westfield
Ayers, Osborn
Corbett, Clifton S.
Knoll, Alexander F.
Schaub, Stanley J.
Woodbridge
McLellan, Roy D.
Woodstown
Brennecke, Henry M.

NEW MEXICO

Albuquerque
Albright, James Lofton
Buckwold, S. Joshua
Purtymun, William D.
Rosenzweig, A.
Schulze, Jack D.
Farmington
Spencer, Charles W.
Grants
Coudray, R. M.
Las Cruces
Barrett, Richard L.
Los Alamos
Douglass, Robert M.
Kempter, Charles P.
Santa Fe
Holterman, Leslie W.
McCaig, Donald W., Jr.
Renault, Jacques R.
Wright, Walter H.
Silver City
Schmitt, Harrison Ashley
Socorro
Budding, Antonius J.
Maher, Frank L.
Sun, Ming-Shan
Werner, Robert W.
University Park
Anderson, James U.
Shuman, Robert C.
Tamimi, Yusuf N.

NEW YORK

Albany
Isaacs, Thelma
Alfred
Frechette, V. D.
Schreiber, Edward
Takahashi, Taro
Almond
Brownell, Wayne E.
Ashville
Anderson, Daniel H.
Astoria
Soeder, Gustave
Bayside
Lowe, Kurt E.
Belle Harbor
Biren, Helen A.
Brooklyn
Cohen, Charles
Fankuchen, I.
Kurtz, Jacques A.
Segeler, Curt G.
Valentine, Wilbur G.
Buffalo
Burgess, Gordon H.
Flanigen, Edith M.
Glass, Robert K.
Mumpton, Fred A.
Burnt Hills
De Vries, Robert C.
Corning
Alper, Allen M.
Begley, Edward R.
Bush, Edward A.
Kennedy, Edward
McNally, Robert N.
Ribbe, Paul H.
Van Cott, Harrison C.
Elmira
Buck, Daniel C.
Endicott
Roberson, Herman E.
Falconer
Wagner, James B.
Flushing
Gold, Winston
Moed, Irwin
Renfro, George E.
Glens Falls
Rowley, Elmer B.
Hamilton
Trainer, D. W., Jr.
Hollis
Dunne, James A.
Raisch, William
Irvington-on-Hudson
Parrish, William
Ithaca
Anderson, Alfred L.
Burfoot, J. Dabney, Jr.
Christman, Robert A.
Katonah
Davis, Donald W.

- Kenmore
  - Armistead, Donald A.
  - Awald, Clifford J.
  - Hagglove, Howard J.
  - King, John S.
- Lewiston
  - Robson, Homer L.
- Little Falls
  - Hurley, Donal
- Malverne
  - Rohl, Arthur N.
- Mamaroneck
  - Seaman, David M.
- Mineola
  - Kantrowitz, Irwin H.
- Mohegan Lake
  - Pinkus, Albert S.
- Monroe
  - Jaffe, Howard W.
- Newburgh
  - Desmond, Thomas C.
- New Paltz
  - Heyl, George R.
- New Rochelle
  - Margolis, Davis
- New York
  - Abdel-Gawad, A. M.
  - Alexander, A. E.
  - Barrington, Jonathan
  - Bassett, William A.
  - Behre, Charles H., Jr.
  - Bernheimer, Alan W.
  - Blatt, Benjamin C.
  - Cargille, R. P.
  - Chang, Ping Hsi
  - Efremov, Nicholas
  - Foley, William E.
  - Ford, Hugh A.
  - Grahl, Harry
  - Hardy, John A.
  - Herz, Norman
  - Holmes, Ralph J.
  - Horen, Arthur
  - Kamhi, Samuel R.
  - Kerr, Paul F.
  - Lewis, Richard W., Jr.
  - Martin, E. B.
  - Mason, Brian H.
  - Matzko, John J.
  - Maxwell, Charles H.
  - Merritt, Phillip L.
  - Molloy, Martin W.
  - Montgomery, Arthur
  - O'Connell, Daniel T.
  - Parker, Alfred B.
  - Pierson, Charles T.
  - Poldervaart, Arie
  - Pough, Frederick H.
  - Prinz, Martin
  - Sampter, E. L.
  - Singewald, Quentin D.
  - Smith, Raymond J.
  - Tilson, Seymour
  - Trautz, Otto R.
  - Walker, Alan
- Palisades
  - Kulp, J. Laurence
- Pearl River
  - Horowitz, Ross M.
- Peekskill
  - Zodac, Peter
- Pelham
  - Weidhaas, Ernest
- Pittsford
  - Alling, Harold L.
  - Buckner, Dean A.
  - Jensen, David E.
- Port Chester
  - LaGanza, Richard F.
- Port Washington
  - Platt, Philip L.
- Poughkeepsie
  - Chamberlain, Barbara
- Riverhead
  - Taylor, Harold A., Jr.
- Rochester
  - Cronin, James B.
  - Pinch, William W.
  - Van Horn, Maurice H.
  - Wishart, James S.
- Rye
  - Miller, F. Stuart
- Saratoga Springs
  - Strock, Lester W.
- Schenectady
  - Smith, Edward S. C.
- Staten Island
  - Wellnitz, Anna M.
- Syracuse
  - Chute, Newton E.
  - de Waard, Dirk
  - Hall, F. P.
  - Yorks, Kenneth P.
- Tonawanda
  - Wolkodoff, V. E.
- Troy
  - Kolaian, Jack H.
  - Rosenholtz, Joseph L.
  - Smith, D. T.
- Tuxedo
  - Selchow, Donald H.
  - Zwicker, Walter K.
- Westbury
  - Dorne, Arthur
- Williamsville
  - Norton, Dorita A.
- Yonkers
  - Moyd, Louis

NORTH CAROLINA

- Asheville
  - Wadewitz, Wilhelm M.
- Belmont
  - Hanahan, John, Jr.
- Chapel Hill
  - Maurice, Charles S.
- Durham
  - Furbish, William J.
- Greenville
  - Read, Floyd M., Jr.
- Murphy
  - Van Horn, Earl C.
- Raleigh
  - Davis, Harry T.
  - McCracken, Ralph J.
  - Nowlan, W. J.
  - Stuckey, J. L.
- Winston-Salem
  - Perloff, Louis

NORTH DAKOTA

- Bismarck
  - Bell, Gordon L.

OHIO

- Akron
  - Cook, Kathryn H.
- Ashtabula
  - Henderson, Barry K.
- Barberton
  - Borgen, James M.
  - McCreery, G. L.
- Brecksville
  - Bersticker, Albert C.

Hawisher, Thomas H.

Canton
Poole, S. W.

Cincinnati
Cameron, Thomas B., Jr.
Pash, Edgar A.
Peters, Gerald A.
Scherrer, Peter
Stilwell, Stephen N.
Thomas, Everett A.
White, J. F.

Cleveland
Chace, Fred M.
Holden, Guerdon S.
Lammers, George E.
Lee, Harley C.
Mielenz, Richard C.
Ray, James A.
Sara, Raymond V.
Shurtz, Robert F.
Specker, Robert A.
Weitz, John H.

Cleveland Heights
Cook, William R., Jr.

Columbus
Coldwell, Samuel W.
Coleman, Leslie C.
Ehlers, Ernest G.
Foster, Wilfrid R.
Gentile, Anthony L.
Gielisse, Peter J. M.
Gomez, Herbert J.
Kurbatov, J. D.
McCaughey, Wm. J.
McConnell, Duncan
Rockett, Thomas J.
Saini, Gulshau R.
Schoenlaub, R. A.
Sclar, Charles B.
Smith, William Lee
Wenden, Henry E.

Dayton
Koopman, Donald E.

Delaware
Bassett, Allen M.
Shanklin, Robert E.

Euclid
Olsen, Edward J.

Lakewood
Holladay, Curtis O.

Millerburg
Drushell, Herbert T.

Newark
Erickson, Thomas D.
Scheffler, Lewis F.

Oxford
Bever, James E.
Brock, William W.
Passero, Richard N.
Scotford, David M.
Thoms, John A.

Tiffin
Brant, Arthur M.

Toledo
Eitel, Wilhelm
LaLonde, Ronald E.

Warren
Cline, Stanley H.

Wooster
Mann, Charles F.

OKLAHOMA

Duncan
Leffler, I. D.

Norman
Lucas, Elmer L.
Mankin, Charles J.
Merritt, C. A.

Ponca City
Bohor, Bruce F.
Knutson, Carroll F.
Weaver, Charles E.

Tulsa
Enlows, Harold E.
Frederickson, A. F.
Friedman, Gerald M.
Kidwell, Albert L.
Kornfeld, Joseph A.
Leighton, Morris W.
McLaren, Eugene H.
Murray, Albert N.
Watkins, Kenneth

OREGON

Albany
Lynde, Harold W., Jr.

Camp White
Binker, Elmer J., Jr.

Corvallis
Hess, Harold D.
Kauffman, A. J., Jr.
Trumpour, Harry J.

Eugene
Lund, Ernest H.
McGuinness, Albert L.
Staples, Lloyd W.
Waters, A. C.

Milwaukie
Bell, James F.

Portland
Bowen, Richard G.
Dake, H. C.
Grubaugh, Philip L.
Keen, Albert J.
Lutton, Richard J.
Rahko, Hannes

Salem
Morley, Russell A. E.

Springfield
Gile, Virgil K.

PENNSYLVANIA

Abington
Trudell, Harry W.

Ardmore
Hamilton, Charles C.

Bellefonte
Sarver, James F., Jr.

Bethlehem
Fraser, Donald M.
Fretz, A. H.
Gault, H. R.
Grant, Raymond W.

Bryn Mawr
Watson, E. H.

Butztown
Erickson, Edwin S., Jr.

Chadds Ford
Young, Howard S.

Doylestown
Evans, Harold

Erie
Oldenburg, John A.

Gettysburg
Olinger, Barton W.

Gwynedd Valley
Presgrave, Cyril

Harrisburg
Lapham, Davis M.
Socolow, Arthur A.

Irwin
Snook, Joseph M.

- Lansdale
  - Belz, Charles A.
- Laureldale
  - Myers, David K.
- Levittown
  - Regis, Andrew J.
  - Williams, James M.
- Malvern
  - Fishwick, John H.
- Monroeville
  - Dodge, Nelson B.
  - Gray, Ralph J.
  - Snow, Ronald B.
- Narberth
  - Heller, John L.
- Natrona Heights
  - Willems, H. W. V.
- New Kensington
  - Ricker, R. W.
- Norristown
  - Mikami, Harry M.
- Oxford
  - Gillingham, Thomas E.
- Philadelphia
  - Crabb, Charles C.
  - Davies, M. Willard
  - Jaffe, Arthur
  - Kissileff, M. Z.
  - Knabe, William C.
  - Metz, Edward A.
  - Patterson, A. L.
  - Schleifer, Ronald H.
  - Stephano, C. S.
  - Sutton, Willard H.
  - Travis, David
  - Venuto, C. Joseph
  - Wherry, Edgar T.
- Pittsburgh
  - Berman, Robert
  - Carter, Forrest L.
  - Cohen, Alvin J.
  - Crump, Robert M.
  - Deul, Maurice
  - Earley, J. W.
  - Ghose, Subrata
  - Gillery, F. H.
  - McVeagh, William J.
  - Milne, Ivan H.
  - Pelto, Chester R.
  - Pollack, Sidney S.
  - Scott, Robert K.
  - Shimamoto, Mrs. K.
  - Smoot, Thomas W.
  - Strickler, Donald W.
  - Van Tassel, Harry C.
  - Wells, Ralph G.
- Plymouth Meeting
  - Sidler, Aubrey G.
- Rosemont
  - Layman, Frederic G.
- Sharon
  - Wilson, Lorenz H.
- Springfield
  - Albrecht, Herbert O.
- State College
  - Bogardus, E. Hal
  - Brown, William L.
  - Cassidy, William A.
  - Cohen, Howard M.
  - Corrigan, Donald J.
  - Dachille, Frank
  - Ridge, John D.
  - Roy, Della
  - Saha, Prasenjit
  - Woodtli, Robert
  - Wright, Harold D.
- University Park
  - Bates, Thomas F.
  - Brindley, G. W.
  - Burnham, C. Wayne
  - Dolsen, C. Philip
  - Fawcett, James J.
  - Freund, Friedemann F.H.
  - Griffiths, John C.
  - Hoffer, Abraham
  - Hummel, Floyd A.
  - Jeffries, Charles D.
  - Johnson, Leon J.
  - Keith, Mackenzie L.
  - Krynine, Paul D.
  - Osborn, E. F.
  - Pepinsky, R.
  - Ramspott, Lawrence D.
  - Rosenberg, Philip E.
  - Roy, Rustum
  - Smith, Chester M., Jr.
  - Smith, Joseph V.
  - Tuttle, O. F.
  - Walter, Louis S.
  - Warshaw, Charlotte M.
  - Williamson, William O.
- West Chester
  - Bryant, W. M. D.
  - Morgan, Paul W.
- Williamsport
  - Speckmann, Ronald G.
- Wrightsville
  - Duersmith, L. J.

RHODE ISLAND

- Kingston
  - Miller, Clarence E.
- Providence
  - Eckelmann, F. Donald
  - Quinn, Alonzo W.
  - Ross, Virginia F.
- Rumford
  - Sherman, Walter C.
- Westerly
  - Gibney, Thomas M.

SOUTH CAROLINA

- Columbia
  - Taber, Stephen

SOUTH DAKOTA

- Custer
  - Brown, Delmer L.
- Huron
  - Howells, Lewis
- Keystone
  - Morgan, Jennings D.
- Rapid City
  - Rapp, George, Jr.
  - Roberts, W. L.

TENNESSEE

- Kingsport
  - Sievers, D. C.
- Knoxville
  - Harper, Delbert D.
  - Harris, Lawrence A.
  - Johnson, William M.
  - Kopp, Otto C.
  - Manus, Ronald W.
  - Milici, Robert C.
  - Moneymaker, Berlen C.
  - Palmer, Raleigh A.
- Nashville
  - Rankin, Douglas W.
- Norris
  - Cotter, Perry G.
  - Denny, M. V.
  - Gibbs, Gerald V.

Shell, Haskill R.
Oak Ridge
Clark, G. Wayne
Tamura, Tsuneo
Wittels, Mark C.
Tullahoma
Langford, Lawrence

TEXAS

Austin
Barnes, Virgil E.
Brown, Thomas E.
Bullard, Fred M.
Clabaugh, Stephen E.
Illich, Harold A.
Ingerson, Earl
Jonas, Edward C.
King, Elbert A., Jr.
Lindhe, Henry E.
Lonsdale, John T.
McCutchen, William T.
McIntire, William L.
Messina, Mario L.
Simonsen, Stanley H.
Snyder, John L.
Stone, Robert L.
Weiss, E. J.
White, John W.
Beaumont
Foss, Ted H.
Bellaire
Dahl, Harry M.
College Station
Blank, Horace R.
Kunze, George W.
Conroe
Silberstein, M. L.
Dallas
Brown, H. Trueheart
Riggs, Karl A., Jr.
El Paso
Dillon, William E.
Fort Worth
Ehlmann, Arthur J.
Perry, John L.
Freeport
Levinson, Alfred A.
Harlingen
Roddy, David J.
Houston
Altman, Thomas D.
Burst, John F.
Carman, Max F., Jr.
Clarke, Thomas G.
Deffeyes, Kenneth S.
Donnelly, Thomas W.
Greenwood, Robert
Halbouty, Michel T.
Hsu, K. J.
Lewis, Donald R.
Pliler, Richard
Richey, Charles I.
Rogers, John J. W.
Rolshausen, F. W.
Rowland, Richards A.
Shaw, Daniel B.
Slovinsky, Raymond L.
Sowers, George M.
Steinfink, Hugo
Studer, Heinz P.
Taylor, Ralph E.
Irving
Halsey, Jonathan H.
Lubbock
Mattox, Richard B.
Sturm, Edward
Wade, F. Alton
Midland
Kelley, Dana R.
Perusek, Cyril J.
Waldschmidt, W. A.
San Antonio
Orchard, C. D.
Tyler
Drummond, Paul L.
Roberts, Oran M.
Waco
Huang, Walter T.
McAtee, James L., Jr.

UTAH

Devils Slide
Irwin, E. Glen
Midvale
Vaughan, Allan C.
Moab
Steen, Charles A.
Price
Burge, Donald L.
Provo
Bullock, Kenneth C.
Owens, Joseph
Phillips, William R.
St. George
Wyman, Richard V.
Salt Lake City
Anderson, Barbara J.
Condie, Kent C.
Crawford, Arthur L.
Kapantais, George G.
Lewis, Charles F.
Miller, Roswell, III
Odekirk, Jerry R.
Ray, Otto A.
Reeves, Augustus
Rose, Arthur W.
Stringham, Bronson
Taylor, Allen O.
Viksne, Andris
Whelan, James A.

VERMONT

Burlington
Doten, Robert K.
Montpelier
Chidester, Alfred H.

VIRGINIA

Alexandria
Clarke, James W.
Arlington
Chao, Edward C. T.
Shaw, Herbert R.
Sinkankas, John
Wones, David B.
Blacksburg
Hallinger, Donald E.
Kane, John K., II
Michael, Carlton A.
Redden, Jack A.
Charlottesville
Calver, James L.
Edmundson, Raymond S.
Farmer, George T., Jr.
Hobbs, Bruce
Mitchell, Richard S.
Pegau, Arthur A.
Young, Robert S.
Falls Church
Bastron, Harry
Foster, William J.
Lynchburg
Duplin, V. J., Jr.

McLean
- Corwin, Gilbert

Pearisburg
- Bryan, James W.

Richmond
- Dreyer, Robert M.

Wallace
- Fagan, James M.

Waynesboro
- Campbell, Frank H.

WASHINGTON

Ephrata
- Baumann, H. N.

Lynnwood
- Haggard, L. R.

Olympia
- Thorsen, Gerald W.
- Valentine, Grant M.

Pullman
- Campbell, Charles D.
- Kittrick, James A.
- Kjelland, Charles
- Sorem, Ronald K.

Renton
- Takatsui, Fumio

Richland
- Ames, Lloyd L., Jr.
- Pearce, D. W.

Seattle
- Barksdale, Julian D.
- Ellis, Ross C.
- Fuller, Richard E.
- Goodspeed, George E.
- McKee, E. Bates, Jr.
- Misch, Peter
- Sauers, Jack D.
- Schmidt, Dwight L.
- Vance, Joseph A.

Spokane
- Canwell, James A.
- Fair, Karl C.
- Hetland, Donald L.
- McGarry, Fred M.
- Spencer, Harvey W. J.
- Vhay, John S.

Tekoa
- Burson, G. Kent

WISCONSIN

Beloit
- Calland, Richard H.

Fond du Lac
- Boehnlein, Wallace J.

Madison
- Bailey, S. W.
- Brown, Bruce E.
- Cameron, Eugene N.
- Emmons, R. C.
- Gates, Robert M.
- Jackson, M. L.
- Mitra, S. P.
- Ostrom, Meredith E.
- Wilsman, Lee J.

Milwaukee
- Knoske, Gene

Sheboygan
- Hinz, George C.

West Allis
- Price, Eugene W.

Williams Bay
- Stenstrom, Richard C.

WYOMING

Laramie
- Fisk, H. G.
- Parker, Ronald B.
- Short, Ben L.

Rawlins
- Grace, John D.

Riverton
- Woo, Ching Chang

Wapiti
- Coe, Darrell L.

PUERTO RICO

Santurce
- Grossman, I. G.

OTHER COUNTRIES

ALGERIA

Algiers
- Royer, L.

ARGENTINA

Adrogue
- Villar Fabre, Jorge Felix

Buenos Aires
- Galloni, Ernesto
- Gonzalez-Bonorion, F.
- Linares, Enrique
- Mortola, Edelmira

San Miguel de Tucuman
- Bossi, Gerardo E.
- Porto, Juan C.

Santa Fe
- Benet, Jose Luis

Temperley
- Novitzky, Alejandro

AUSTRALIA

Adelaide
- Kleeman, A. W.

Broken Hill
- Baker, William E.

Canberra
- Chappell, Bruce W.
- Morgan, William R.

Carlton
- McAndrew, John

Cooma
- Lafeber, David
- Sharp, Kenneth R.

Highett
- Segnit, E. R.

Kalgoorlie
- Ostwald, Joseph

Kensington
- Loughnan, F. C.

Lockleys
- Sweatman, Thomas R.

Melbourne
- Edwards, A. B.
- McLaughlin, R. J. W.

Nedlands
- Prider, Rex T.

North Melbourne
- Mawby, M. A.

Redbank
- Sauer, Kevin S.

Sydney
- Chaffer, Judith A. F.
- Hamilton, John D. G.

  - Vallance, Thomas G.

AUSTRIA

- Knappenberg
  - Meixner, Heinz
- Vienna
  - Machatschki, F.
  - Scharbert, Heinz G.

BELGIUM

- Brussels
  - Bartholome, Paul
- Ghent
  - Schoep, A.
- Louvain
  - Moreau, Jules F.
- Wieze
  - Witte, George De
- Woluwe
  - Buttgenbach, Henry J.

BOLIVIA

- La Paz
  - Ahlfeld, Federico
  - Murillo, Carlos V.
  - Reyes, Jorge Munoz
  - Zubrzycki, Poitr
- Oruro
  - Berndt, Fritz

BRITISH SOLOMON ISLANDS

- Honiara
  - Thompson, R. B.

BRAZIL

- Belem
  - Butler, Robert D.
- Belo Horizonte
  - Guimaraes, Djalma
- Recife
  - Da Silva, Rilson R.
  - Ebert, Heinz
  - Loureiro, Ivan A.
  - Rao, A. Bhaskara
  - Stark, John T.
- Rio de Janeiro
  - Tavora, Elysiario
  - Vieira, Pedro Albertino
- Salvador
  - Allard, Gilles
  - Leo, Gerhard W.
- Sao Paulo
  - Campos, João, E. S.
  - Franco, Rui Ribeiro
  - Lowenstein, Walter
  - Pieper, Bernhard T.
  - Porto, Murillo Cabral

CANADA

Alberta

- Calgary
  - Beveridge, Alexander J.
- Edmonton
  - Campbell, Finley A.
  - Hitchon, Brian
  - Peterman, Zell E.

British Columbia

- Chapman Camp
  - Freeze, Arthur C.
- Holberg
  - Lincoln, Earle
- New Westminster
  - Gower, John A.
- Port Coquitlam
  - Bennie, Archibald F.
- Vancouver
  - Campbell, Douglas D.
  - Danner, Wilbert R.
  - Hyndman, Donald W.
  - Thompson, Robert M.
- Wilson Creek
  - Forbes, Alistair

Manitoba

- Winnipeg
  - Ferguson, R. B.

New Brunswick

- Fredericton
  - MacKenzie, G. S.

Nova Scotia

- New Glasgow
  - Goodman, Nordau R.

Ontario

- Chippawa
  - Finlay, G. R.
- Cobalt
  - Thomson, Robert
- Copper Cliff
  - Harcourt, George A.
- Don Mills
  - Graham, A. R.
  - Slavik, Frank H., Jr.
- Downsview
  - Spooner, Charles M.
  - Woda, George R.
- Falconbridge
  - Mutch, A. D.
- Fort Frances
  - Shapiro, Leo, Jr.
- Hamilton
  - Edgar, Alan D.
  - Stevens, Robert P.
- Kingston
  - Berry, Leonard G.
  - Findlay, David C.
  - Hawley, James E.
  - Jolliffe, A. W.
- Lakefield
  - Deane, Rodney W.
- Lively
  - Turja, Oscar
- London
  - Boone, Gary M.
  - Kramer, James R.
- Manotick
  - Prince, Alan T.
- Markham
  - Scott, Douglas
- Ottawa
  - Barnes, William H.
  - Brydon, James E.
  - Buchanan, Robert M.
  - Forman, S. A.
  - Haycock, Maurice H.
  - Hogarth, Donald D.
  - Hughson, Michael R.
  - Jenness, Stuart E.
  - Kaiman, S.
  - MacLaren, Alexander S.
  - Nickel, Ernest H.
  - Robinson, S. C.
  - Smith, Charles H.
  - Spence, H. S.
  - Stevens, Robert D.
  - Traill, R. J.
  - White, William E.
- Rosebank Station
  - MacKeracher, D. M.
- Sarnia
  - Rowe, Robert B.

- Toronto
  - Davidson, Stanley C.
  - Dolar-Mantuani, L.
  - Ebbutt, Frank
  - Fraser, H. J.
  - Gorman, D. H.
  - Johnston, Robert A. A.
  - Mandarino, Joseph A.
  - Meen, V. Ben
  - Michener, Charles E.
  - Moddle, Douglas A.
  - Moore, E. S.
  - Murphy, Richard F.
  - Nuffield, Edward W.
  - Seward, Terry M.
  - von Bitter, Peter

Quebec

- Arvida
  - Gummer, W. K.
  - Sabiston, Gordon H.
- Chibougamau
  - Vollo, Nels B.
- Chicoutimi
  - Tremblay, Abbé J. A.
- Granada
  - St. Louis, Aurele
- McMasterville
  - Matthews, Fred W.
  - McIntosh, Alex O.
- Montreal
  - Frueh, Alfred J., Jr.
  - Perrault, Guy
  - Stevenson, John S.
  - Stevenson, Mrs. John S.
- Outremont
  - Graham, R. P. D.
- Quebec
  - Beland, Rene
  - Girault, J. P.
  - Remick, Jerome H.
- Ste Anne-de-la-Pocatiere
  - Ledoux, Robert L.

Saskatchewan

- Regina
  - Pearson, Walter J.
- Saskatoon
  - Arnold, Ralph G.
  - Smith, James R.

CHILE

- Potrerillos
  - Hunt, John P.
- Santiago
  - Aguirre L., Luis
  - Aliste Torres, Nelson
  - Alvarez Schultz, Leonardo
  - Bravo Santander, Nelson
  - Mehech Numan, Sonia
  - Oritz, Francisco J.

COSTA RICA

- San Jose
  - Durando, Ottavio G.

CUBA

- Havana
  - Cuervo, A. America Ana
  - Samuels, Fred
  - Wandke, A. D.

CZECHOSLOVAKIA

- Bratislava
  - Kamenicky, Jakub
  - Matherny, M. N.
- Kutna Hora
  - Novak, Frantisek
- Praha
  - Bernard, Jan
  - Bouska, Vladimir
  - Cech, Frantisek
  - Konta, Jiri
  - Kutina, Jan
  - Padera, Karel
  - Slansky, Ervin
  - Zak, Lubor

DENMARK

- Copenhagen
  - Clausen, Hans

EGYPT

- Alexandria
  - Gindy, Amin R.
  - Hilmy, M. E.
- Cairo
  - Higazy, Riad A.

ENGLAND

- Birmingham
  - Adie, Raymond J.
  - Lacy, Ernest D.
- Cambridge
  - Agrell, S O.
  - Gay, Peter
  - Henry, N. F. M.
  - LeBas, Michael J.
  - Le Maitre, R. W.
  - Megaw, Helen
  - Nockolds, S. R.
  - Taylor, W. H.
  - Tilley, C. E.
  - Wood, John A., Jr.
- Durham
  - Dunham, K. C.
  - Emeleus, Charles H.
  - Harbord, Norman H.
  - Nesbitt, Robert W.
  - Phillips, Roy
- Englefield Green
  - Sunagawa, Ichiro
- Harpenden
  - Brown, George
- Hathersage
  - Nagelschmidt, G.
- Horley
  - Bannister, F. A.
- Leeds
  - Cox, E. G.
  - Harris, Peter G.
  - Hornung, George
  - Kennedy, W. Q.
  - Knorring, Oleg Von
  - Wyllie, P. J.
- London
  - Anderson, B. W.
  - Andrew, Gerald
  - Bowie, Stanley H. U.
  - Claringbull, G. F.
  - Dearnley, Raymond
  - Dunning, Frederick W.
  - Elliot, Ranald W.
  - Embrey, Peter G.
  - Hallimond, A. F.
  - Horne, J. E. T.
  - Lawson, Robert I.
  - Lonsdale, Kathleen

McKie, Duncan
Millman, A. P.
Sabine, P. A.
Young, Brian R.

Manchester
Bradshaw, Neville
Howie, R. A.
Mackenzie, William Scott
Zussman, Jack

Newcastle
Simcock, J. H.

Oxford
Bown, Michael G.
Brown, George Malcolm
Kingsbury, A. W. G.
Vincent, E. A.
Wager, L. R.

Reading
Russell, Sir Arthur

Rotherham
Cockbain, Alan G.

St. Albans
Chambers, G. P. C.

Virginia Water
Seager, A. F.

Watford
Midgley, H. G.

West Hartlepool
Ashford, John H.

Worsley
Buckley, Harold E.

FINLAND

Helsinki
Eskola, Pentii
Lehijärvi, Mauno
Rankama, Kalervo
Sahama, Thure Georg
Savolahti, Antti
Wiik, H. B.

Otaniemi
Marmo, Vladi
Meriläinen, K.
Mikkola, Toini
Neuvonen, K. J.
Vorma, Atso I.

FRANCE

Bellevue
Rose, J.

Maizieres-Les-Metz
Sokoloff, Vsevolode

Paris
Blondel, F.
Guillemin, Claude
Letort, Yves
Orcel, J.
Permingeat, Francois
Stugard, Frederick, Jr.
Wyart, J.

GERMANY

Aachen
Vultee von, Joachim

Berlin-Charlottenburg
Strunz, Hugo

Bonn
Scheumann, K. H.

Bovenden
Bultemann, Hans W.

Erlangen
Ernst, T.

Essen
Kirsch, Helmut
Mackowsky, Marie-Therese

Frankfurt
Fischer, Karl
Hahn, Theodor
O'Daniel, H.

Freiburg
Tröger, W. E.

Gottingen
Bachmann, H. G.
Baur, W. H.
Beil, Hanswilhelm
Bolter, Ernst
Chudoba, Karl F.
Correns, Carl W.
Echle, Wolfram
Gattow, G.
Glemser, Oskar
Heydemann, Annerose
Johns, William D.
Kalk, Eberhard K. E.
Koritnig, Sigmund Ch.
Lippmann, Friedrich
Lohmann, Ludwig
Niemann, Hans
Ricke, Werner
Wedepohl, Hans
Zemann, Josef

Hamburg
Brendler, W.
Lietz, J. H. C.

Hannover
Eckhardt, Franz-Jörg
Gaertner, Hans R. von
Langer, Michael
Schellmann, Werner

Heidelberg
Ramdohr, Paul

Idar-Oberstein
Schlossmacher, Karl H.
Wild, George O.

Kiel
Hellner, Erwin E.

Lenglern
Hartmann, Martin

Lubeck
Dettmer, Friedrich

Marburg
Althaus, Egon
Brehler, Bruno
Platen, Hilmar V. B. von
Winkler, H.

Munster
Seifert, H.

Neustadt am Rbge
Schmitz, Hans H.

Regensburg
Koster, H. M.

Wietze/Kreis Celle
Hering, Otto H.
Zimmerle, Winfried

Wurzburg
Matthes, Siegfried

GHANA

Obuasi
Dyson, Peter

GREECE

Thessaloniki
Rentzeperis, P. J.

GUATEMALA

Guatemala City
Brineman, John H.
Dengo, Gabriel

HUNGARY

Budapest
- Foldvari, Aladar

ICELAND

Reykjavik
- Thorarinsson, Sigurdur
- Tryggvason, Tomas

INDIA

Bangalore
- Venkayya, E.

Calcutta
- Blade, Lawrence V.

Dharwar
- Sadashivaiah, M. S.

Jammu-Tawi
- Sharma, T. R.

Madras
- Subramaniam, A. P.

Nagpur
- Banerjee, Anil K.

Patna
- Gupta, M. P.

Trivandrum
- Nair, K. V. Krishnan

Waltair
- Krishnan, M. S.

INDONESIA

Bandung
- Bacon, Charles S.

IRAN

Tehran
- Alipour, Keramat

ISRAEL

Jerusalem
- Bentor, Y. K.

Tel-Aviv
- Koppel, B.

ITALY

Caltanisetta
- Ravizzini, E.

Firenze
- Carobbi, Guido

Milano
- Fagnani, Gustavo
- Gramaccioli, Carlo M.

Modena
- Gallitelli, Paolo

Pisa
- Bonatti, Stefano
- Gottardi, Glauco

JAMAICA

Montego Bay
- Hill, V. G.

JAPAN

Fukuoka
- Shirozu, Haruo
- Wada, Koji
- Yamaguchi, Masaru

Gifu-Ken
- Sakuma, Tadashi
- Shiobara, Kanji

Hiroshima
- Toyoda, Hideyoshi
- Umegaki, Yoshiharu

Ibaragi-Ken
- Muto, Tadashi

Kagoshima
- Ishikawa, Hideo

Kawasaki
- Inuzuka, Hideo
- Sugaike, Suezo
- Tanemura, Mitsuro

Kobe
- Yamamoto, Tsutomo

Kurume
- Yamamoto, Hirosato

Kyoto
- Nakagawa, Masao
- Ueda, Tateo

Matsue
- Yokoyama, Kanae

Nagoya
- Ishioka, Kokichi
- Shiozaki, Prof.
- Suwa, Kanenori

Osaka
- Kakitani, Mr.
- Koizumi, Mitsue

Sapporo
- Harada, Zyunpei
- Hariya, Yu
- Ishibashi, Masao
- Togari, Kenji
- Urashima, Yukitoshi

Sendai
- Hasegawa, Shuzo
- Omori, Keiichi
- Yagi, Kenzo

Shizuoka
- Sameshima, Teruhiko

Tobata
- Yamamoto, Takashi

Tokyo
- Aramaki, Shigeo
- Hamachi, Tadao
- Igarashi, Toshio
- Imai, Hideki
- Ito, Tei-Ichi
- Jennings, Charles B.
- Kuno, Hisashi
- Maekawa, Miss Tomoko
- Minato, Hideo
- Morimoto, Ryohei
- Muraoka, Makoto
- Nakahira, Mitsuoki
- Ootsubo, Yoshio
- Saito, K.
- Sakura, Kiyoshi
- Shimazaki, Yoshihiko
- Shimazu, Mitsuo
- Sudo, Toshio
- Takahashi, Hiroshi
- Takeda, Akitsu
- Takeuchi, Yoshio
- Watanabe, Takeo

Tsukuba
- Ichimura, Takeshi

Ube
- Sugaki, Prof.

JUGOSLAVIA

Ljubljana
- Duhovnik, J. M.

KENYA

Nairobi
- Loupekine, I. S.

KOREA

Seoul
- John, Yong Won
- Sang, Kinam

MADAGASCAR

Tananarire
- Behier, J. J. F.

MALAYA

Ipoh
Flinter, Basil
Kuala Lumpur
Hutchison, Charles S.

MEXICO

Mexico City
Arellano, Alberto R. V.
d'Acosta, Julio
Pablo, Liberto de
Fries, Carl, Jr.
Isita, Jose
Knizek, Ian

NETHERLANDS

Arnhem
van der Veen, A. H.
Leiden
Tobi, A. C.
Ypma, Peter J. M.
Wageningen
Doeglas, D. J.

NEW ZEALAND

Dunedin
Carr, Robert M.
Coombs, D. S.
Wellington
Finch, James

NIGERIA

Bukuru
Horn, Aubrey E.

NORWAY

Blommenholm
Sverdrup, Thor L.
Kragero
Johne, C. T.
Oslo
Barth, Tom F. W.
Heier, Knut S.
Nilssen, Borghild
Reitan, Paul H.
Vokes, Frank M.

PAKISTAN

Karachi
Struwe, H.

PERU

Lima
Aguilar Condamarin, Lucio F.
Ayza, Juan
Boggio, Mario Samame
Canessa, Jorge
Miraflores-Lima
Casas, Fernando de las
Oroya
Petersen, Ulrich

PHILIPPINE ISLANDS

Manila
Fernandez, Norberto
Vera, Elpidio C.

PORTUGAL

Coimbra
Correia Neves, Jose M.

PORTUGUESE EAST AFRICA

Lourenco Marques
Oberholzer, Walter

SAUDI ARABIA

Dhahran
Ricketson, Eleanor T.

SCOTLAND

Aberdeen
Glasser, Fred P.
Mackenzie, Robert C.
Phemister, T. C.
Edinburgh
Holmes, Arthur
Stewart, Frederick H.
Phemister, James
Old Aberdeen
Taylor, H. F. W.
Pitlochry
Robertson, Robert H. S.
St. Andrews
Davidson, Charles F.

SOUTH AFRICA

Bloemfontein
deVilliers, J. E.
Cape Town
Sampson, Basil J.
Crown Mines
Nel, Hans J.
Graskop
Skea, Ernest M.
Hatfield Pretoria
Pistorius, Carl W.
Johannesburg
Bawden, F. C. Michell
Hargraves, Robert B.
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Rondebosch
Ahrens, L. H.
Fuller, Arthur O.

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Gwelo
Davey, Bernard
Salisbury
Sergiades, Frank R.

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Grootfontein
Markham, Neville Lawrence

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Folch, Joaquin
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La Coruna
Torre Enciso, Eugenio
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Martinez Strong, Pablo

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Hoganas
Norin, Rolf
Kiruna
Sahlin, Carl Ingemar

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RIPSA
- Eckermann, H. von

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- Uytenbogaardt, W.

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- Mellis, Otto
- Quensel, P. D.
- Wickman, Frans E.

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LAUSANNE
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- Stucker, P.

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- Grunenfelder, Marc
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- Laves, Fritz
- Parker, Robert L.

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MUSOMA P.O.
- Grimmer, Arthur

TURKEY

ISTANBUL
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VENEZUELA

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PUERTO ORDAZ
- Reynolds, Charles D.

WALES

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- Ball, David Francis

CATHAYS PARK
- Wilson, A. J. C.

LLANGENNECH
- Griffiths, Ivor

SHALE'S

LISTING OF FINE BULK MINERALS FOR SCIENTIFIC STUDY PURPOSES

SOLD BY THE POUND

ACTINOLITE (Calif.) Green radiating masses	$ .30 per pound
APATITE (Mexico) Yellow crystals and sections	4.50 per pound
KYANITE (Brazil) Blue crystal blades up to 3"	1.50 per pound
CORUNDUM var SAPPHIRE (Africa) Mostly blue masses & Xl sections	1.25 per pound
CORUNDUM var RUBY (Madagascar) Pink to red Crystal sections	5.75 per pound
BARITE (Calif.) Brown crystals and masses	.50 per pound
MARTITE (Calif.) Black crystal sections and masses	.45 per pound
JET (Utah) Black limb sections and pieces	1.95 per pound
ARAGONITE (Nevada) White stalatitic sections and masses	.45 per pound
KERNITE (Boron, Calif.) White crystal sections and masses	.40 per pound
QUARTZ (Brazil) Crystals and sections w/ inclusion of Phantoms	2.50 per pound
GRIFFITHITE (Calif.) Brown masses	2.50 per pound
BORNITE (Arizona) Pure masses, colorful	1.25 per pound
COLEMANITE (Calif.) Grayish crystals and sections	1.75 per pound
FLUORITE (New Mexico) Green masses	.45 per pound
CHROME ZOISITE with hornblende & Ruby inclusions (Africa)	.95 per pound
PEACOCK COPPER & IRON ORE (Arizona) Irridescent masses	.95 per pound
OPTICAL CALCITE (Mexico) second grade transparent cleavages	2.25 per pound
GEM GRAVELS (Burma) Small gravels containing Spinel, Corundums, etc.	6.00 per pound
TOPAZ (Brazil) Crystal sections and pieces	3.50 per pound
OOLITE (Calif.) Black masses	.35 per pound
TURITELLA AGATE (Wyoming) Brown masses	.45 per pound
IDOCRASE (Calif.) Green masses	.40 per pound
HOWLITE (Calif) White masses some with black web	.45 per pound
JADEITE (Burma) Cut massive pieces showing color, some cores	3.75 per pound
PYRITE (Utah) Crystal sections and masses	.60 per pound
TURQUOISE (Nevada) Mine run vein Turquoise in rock	1.75 per pound
BRAZILIANITE (Brazil) Yellow opaque massive pieces	4.50 per pound
GERMANITE with some Tennantite (Africa) Black massive	20.00 per pound
SPODUMENE (Brazil) Crystals up to 2". White partly gem	7.50 per pound
HANKSITE (Calif.) Crystals up to 2"	1.50 per pound

OUTSTANDING MINERAL SPECIMENS FROM OUR LARGE STOCK

CARNOTITE with some Pitchblende (Emory Co., Utah) One of the largest and richest pieces found. 7x10—weight 15 pounds—$175.00

URANINITE replacing WOOD (Emory Co., Utah) Very rich—2x3—weight 23 ounces—$25.00

TOURMALINE (Himalaya Mine, Mesa Grande, Calif.) New find of fine Specimens. Fine double terminated crystals mostly pink. Approximately 2" long ½" thick—$15.00 and $17.50 each. Single terminated approximately 1" to 1½" very fine pink—$5.00; $7.50 and $10.00 each

PYRITE CRYSTALS (Island of Elba) Beautiful bright crystals. Approximately 2" to 3"—$5.00; $7.50 and $10.00 each

CHRYSOBERYLS (Brazil) Fine mostly complete greenish Crystals. Approximately ½"—$2.00 and $3.00—½"—¾"—$4.00 and $5.00 each

BRAZILIANITE (Brazil) Specimens showing one or more crystal faces. ¾" over 1"—$5.00; $7.50 and $10.00 each

PARTIES INTERESTED IN RECEIVING FREE LISTINGS OF NEW ARRIVALS IN FINE MINERAL SPECIMENS FROM WORLD LOCALITIES PLEASE CONTACT.

SHALE'S

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Los Angeles 35, Calif.
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TRENTON, WISCONSIN, METEORITES

As the supply of meteorites rapidly diminishes with the increasing demand, good examples of siderites, cut, etched and polished to show characteristic Widmanstätten figures are rarely offered for sale. The Trenton siderite (a medium octahedrite) is from one of the oldest known occurrences in this country, the first specimens having been found in 1858. The following slices have been polished and etched to show the characteristic Widmanstätten figures of this important meteorite.

S 33.11	56	gram slab	$ 22.40
S 33.12	75.0	gram slab	30.00
S 33.14	341.0	gram slab	136.40
S 33.15	403.5	gram end piece	161.40
S 33.17	171.0	gram corner piece	68.40
S 33.18	168	gram slab	67.20
S 33.19	182	gram slab	72.80
S 33.20	102.0	gram slab	40.80
S 33.21	187.0	gram slab	75.80
S 33.22	137.0	gram slab	54.80
S 33.23	256.0	gram slab	102.40
S 33.24	246.0	gram slab	98.40
S 33.25	168.5	gram slab	67.40

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Amphibole, var. Nephrite (Jade). A twelve and a half pound chunk of apple green jade, one end sawn, 3 x 8 x 9½″, $60.00 (plus 10% F.E. tax)

Azurite. Arizona. A beautiful royal blue complete cross section of a stalactitic mass, polished on both sides to show fascinating concentric structure 5 x 5½″, $150.00

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Fluorite. England. Choice group of yellow cubic crystals 7½ x 9″, $40.00

Picromerite. Germany. Exceptionally fine crystallized groups, some with halite, anhydrite. 2 x 3″, $5.00; 3 x 4″, $10.00; 4 x 5″, $15.00

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